# Tappi

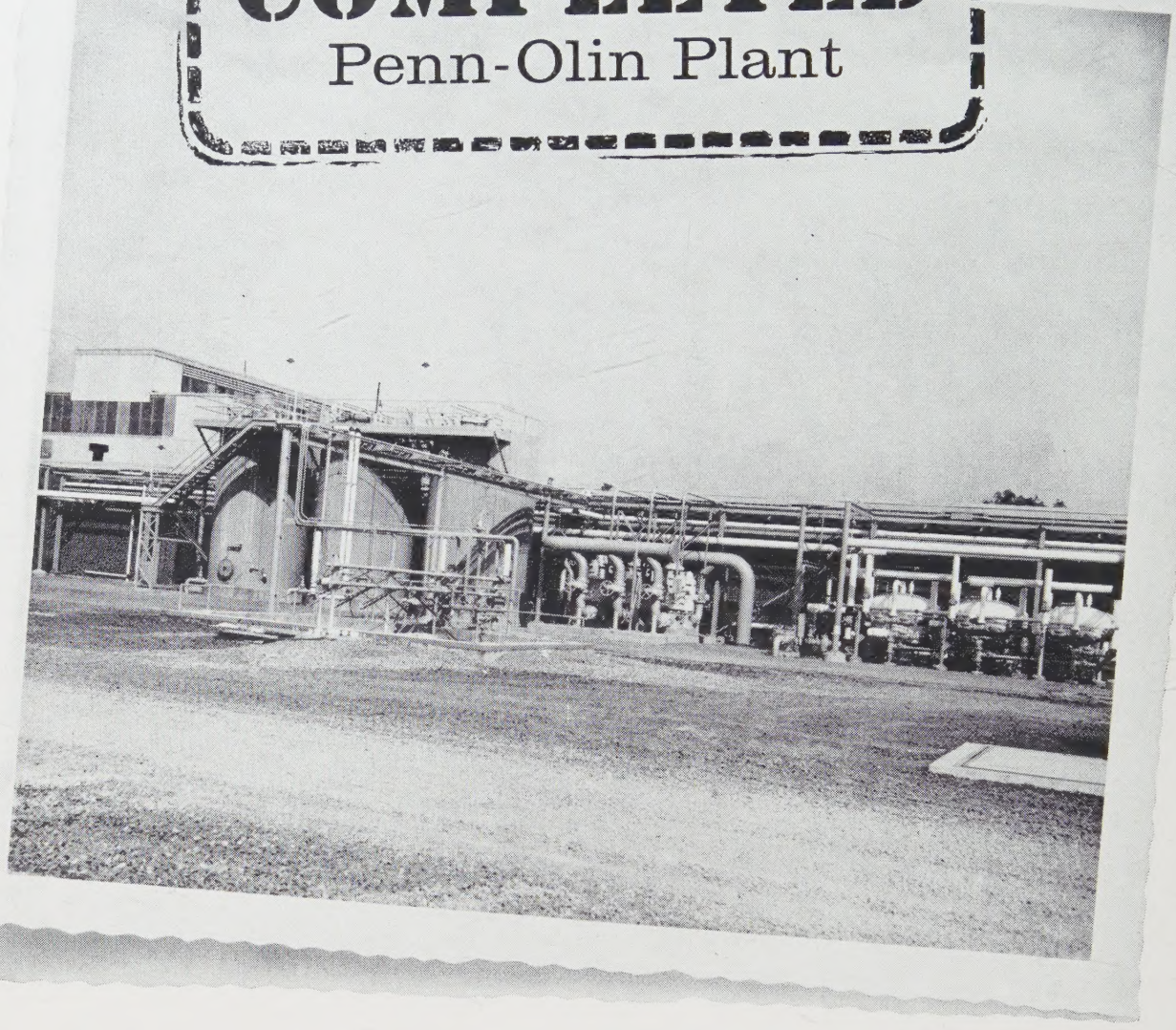
THE JOURNAL OF THE TECHNICAL ASSOCIATION OF THE PULP AND PAPER INDUSTRY

DECEMBER 1961

VOLUME 44 • NUMBER 12

# COMPLETED

Penn-Olin Plant



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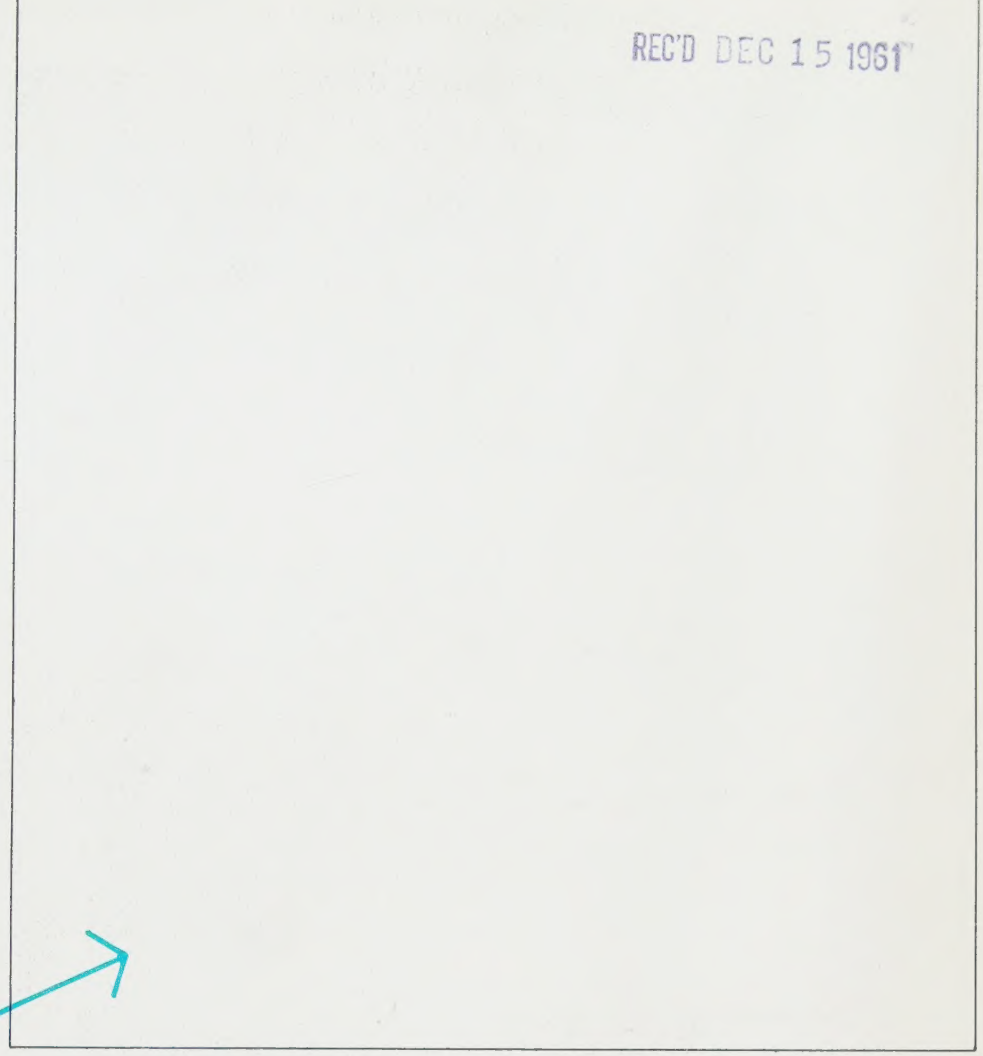
CHEMICALS DIVISION Olin

REC'D DEC 15 1961

# SORRY, THIS IS THE BEST PICTURE WE COULD GET OF POLAR-6



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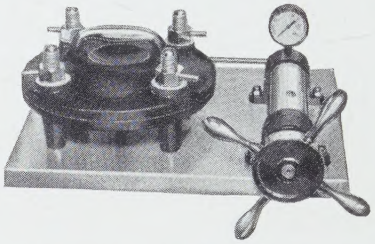
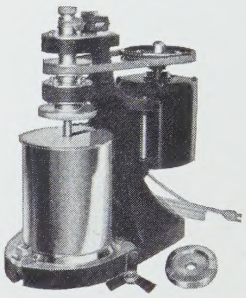


Mounted on 18-8 Stainless steel base. Press on base, or pump only may be purchased separately. (T-205 m)

**TAPPI DISINTEGRATOR**



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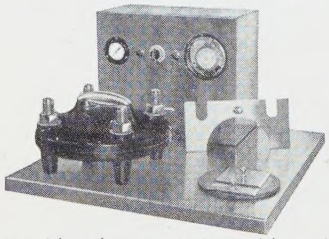

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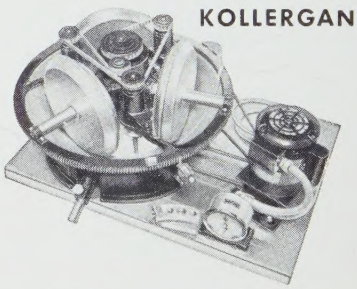

The only instrument that will give truly reproducible beating results. (T-225 sm)

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REC'D DEC 15 1961

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Cameron leadership in roll production research stems from a background which includes the most extensive line of roll production equipment, the most impressive and diversified record of successful experience, and the most abundant reservoir of authentic analytical research data in the field.

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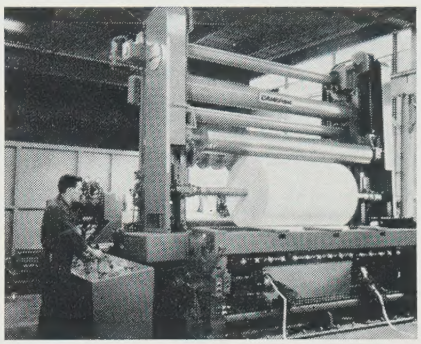
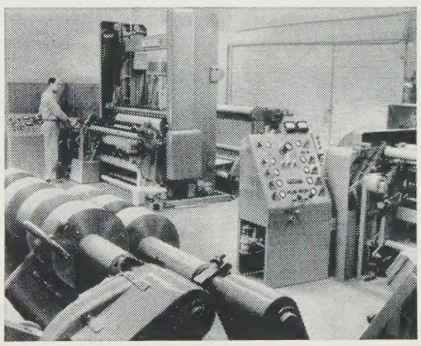
Supported by outstanding plant facilities, Cameron Research has the additional advantage of numerous production and testing devices developed by our own engineers and not available elsewhere. Also, two departments have been fully equipped with two-drum and duplex winding systems, which are employed in engineering development studies and for actual test-runs on all types of materials.

The major interests of Cameron Research are directed toward fundamental advances that will benefit the greatest number of users. Typically, the impact of Cameron integrated unwind-to-rewind roll control has raised competitive standards everywhere. The modern *truly* integrated roll production system is distinctly a Cameron concept, based upon the uniquely comprehensive Cameron background.



An experimental mill type two-drum system, fully equipped for research and development work at Cameron's plant, Dover, N. J.



Another wing of the Cameron Research Service is devoted to a continuous study of converter type duplex and two-drum systems.

Practical applications of Cameron Research have gone far beyond many current standards and pet theories to give Cameron users the reserve capacity, speed, and superb roll quality they need in today's highly competitive markets. Working with many different types of materials, Cameron Research has helped our customers to increase their roll content substantially in large diameter rolls of *superb running quality*. And not with just one cut, but with any required number of cuts across the full width of the web.

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If you are reaching for an unusual achievement in roll production, or if you just want help with a practical production improvement, it will pay you to call on the Cameron Research Service. Write today for more complete information.

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famous TIDLAND pneumatic shafts are sold exclusively through Cameron

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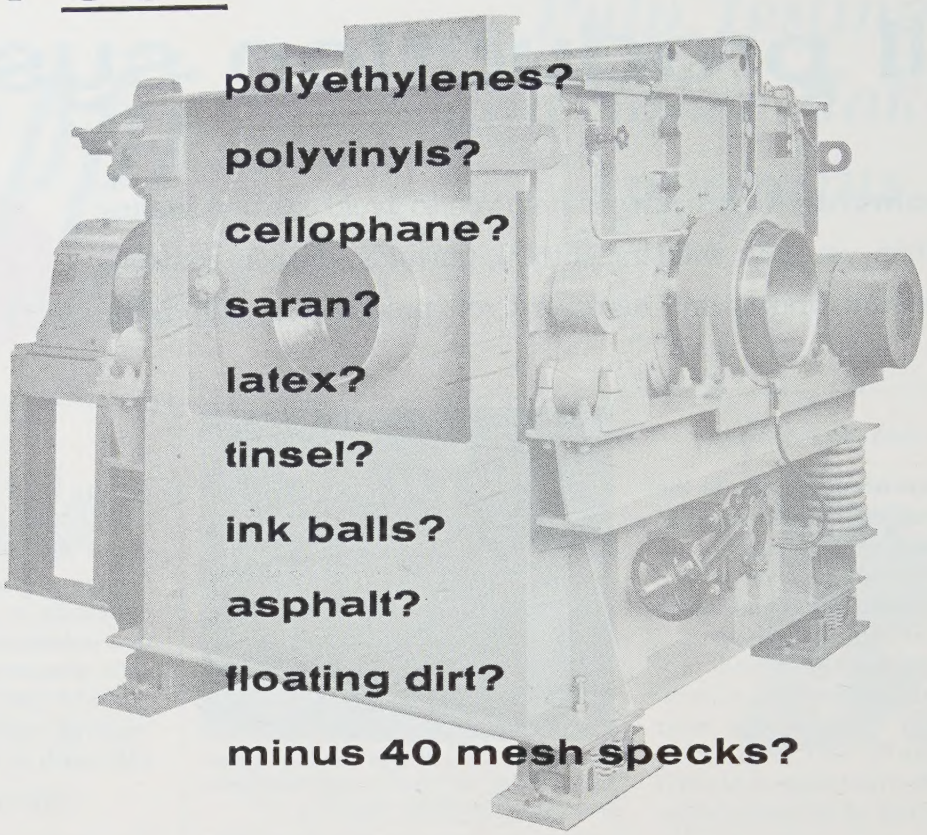
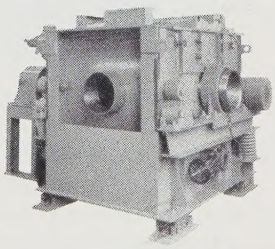



# BIRD VIBROTOR SCREENS



are handling thousands of tons of waste stocks every day. They put large volumes (100 tpd or more) of high consistency stock (2% or more) through fine slotted plates (.012″) to deliver clean, bright fibres free of anything that might cause pinholes or breaks.

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Commodore Hotel, New York, N. Y.
Feb. 18–22, 1962

Thirteenth Coating Conference
Netherland-Hilton Hotel,
Cincinnati, Ohio
May 14–16, 1962

Thirteenth Testing Conference
Benjamin Franklin Hotel,
Philadelphia, Pa.
Sept. 18–20, 1962

Seventeenth Plastics-Paper Conference
Statler-Hilton Hotel, Boston, Mass.
Oct. 8–11, 1962

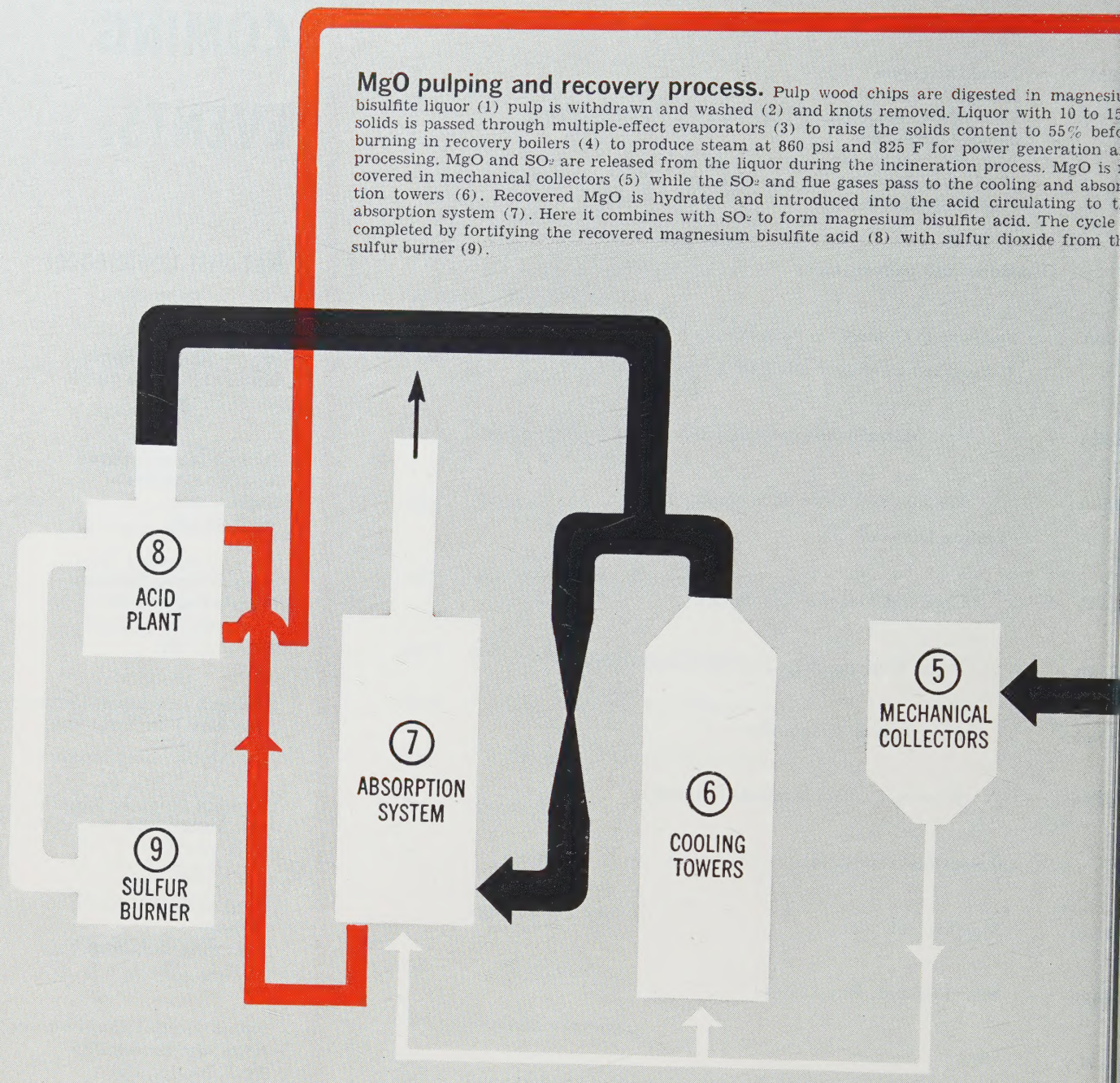
Seventeenth Engineering Conference
Queen Elizabeth Hotel, Montreal, Que
Oct. 14–19, 1962

Twelfth Corrugated Containers Conference
Statler-Hilton Hotel, Boston, Mass.
Oct. 23–26, 1962

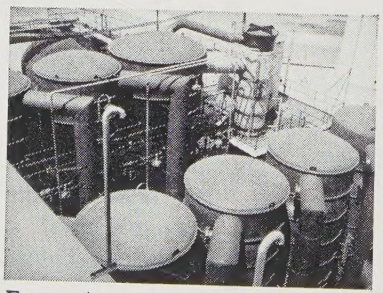
Sixteenth Alkaline Pulping Conference
DeSoto Hotel, Savannah, Ga.
Oct. 31. Nov. 1–2, 1962

**Please refer to Local Section Activities for the locations and dates of Section Meetings**

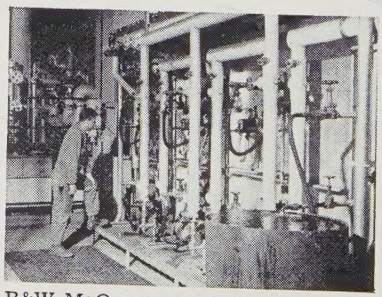
**MgO pulping and recovery process.** Pulp wood chips are digested in magnesiu bisulfite liquor (1) pulp is withdrawn and washed (2) and knots removed. Liquor with 10 to 15 solids is passed through multiple-effect evaporators (3) to raise the solids content to 55% befo burning in recovery boilers (4) to produce steam at 860 psi and 825 F for power generation a processing. MgO and $SO_2$ are released from the liquor during the incineration process. MgO is r covered in mechanical collectors (5) while the $SO_2$ and flue gases pass to the cooling and absor tion towers (6). Recovered MgO is hydrated and introduced into the acid circulating to t absorption system (7). Here it combines with $SO_2$ to form magnesium bisulfite acid. The cycle completed by fortifying the recovered magnesium bisulfite acid (8) with sulfur dioxide from th sulfur burner (9).




# MgO process at Weyerhaeuser's Cosmopo

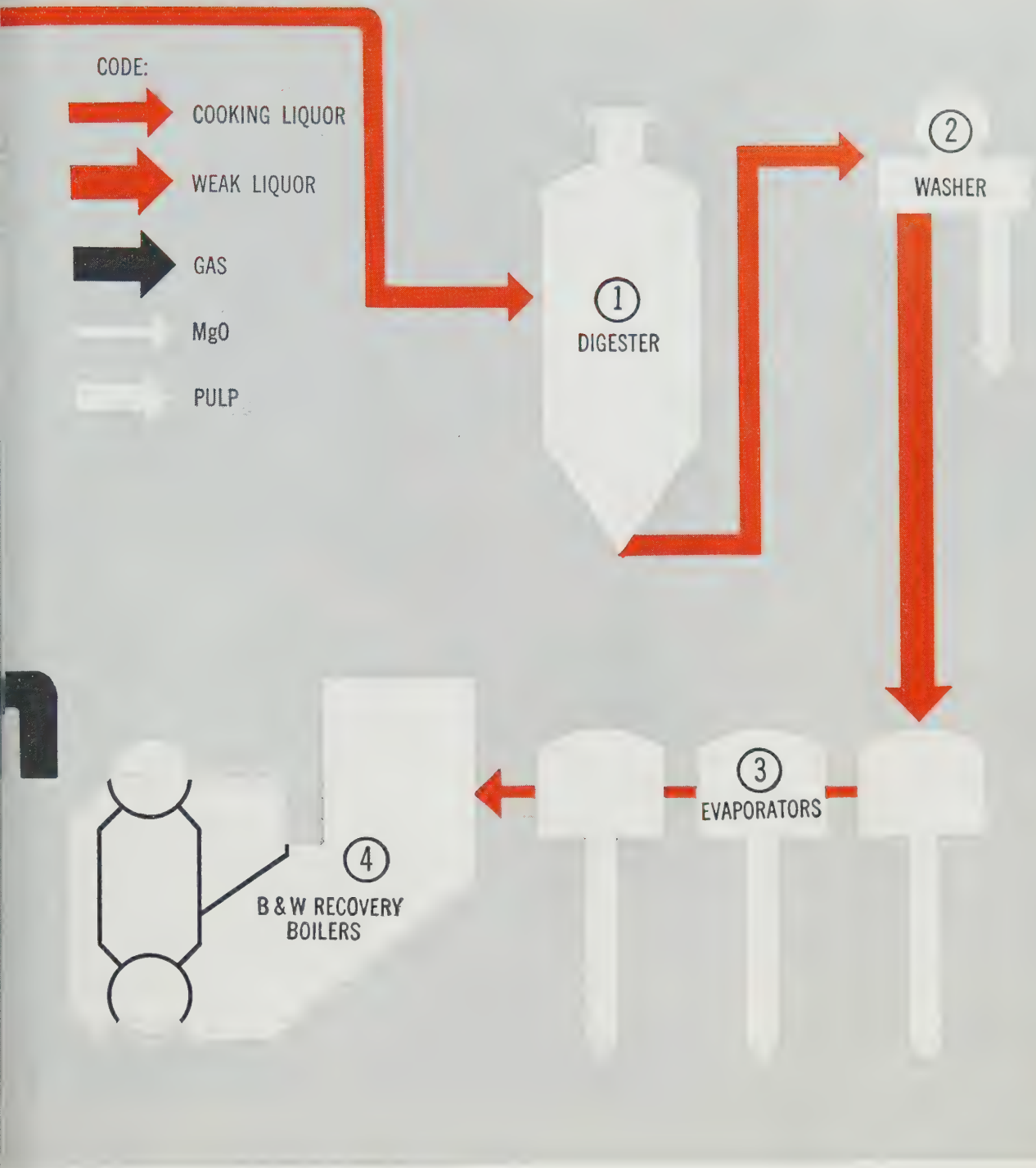


Evaporators in series increase pulping liquor from less than 15% to about 55% solids to permit its burning.



B&W MgO recovery boilers burn the concentrated liquor, supplying 70% of the plant's steam requirements.

Mechanical collector reclaims Mg from furnace flue gas and washes before further use.




# ill recovers 90% of spent liquor solids

Only 20 pounds of magnesium oxide and 40 pounds of sulfur per ton of unbleached pulp are needed for make-up at Weyerhaeuser Company's 400 ton per day sulfite mill at Cosmopolis, Washington.

The simple, closed cycle MgO pulping and recovery process, placed in operation during 1957, reclaims about 90% of the pulping chemicals. The B&W MgO recovery boilers provide 70% of the steam required for the entire plant's power generation and processing needs. A third B&W bark and oil fired boiler provides the balance of steam requirements.

The commercially-proven MgO pulping and recovery process is ideal for either new mills or conversion and modernization of existing mills. Shorter cooking time required for MgO pulping cuts capital and maintenance costs in new installations and permits increasing the production of existing mills. Quality sulfite pulp with high brightness and strength is produced.

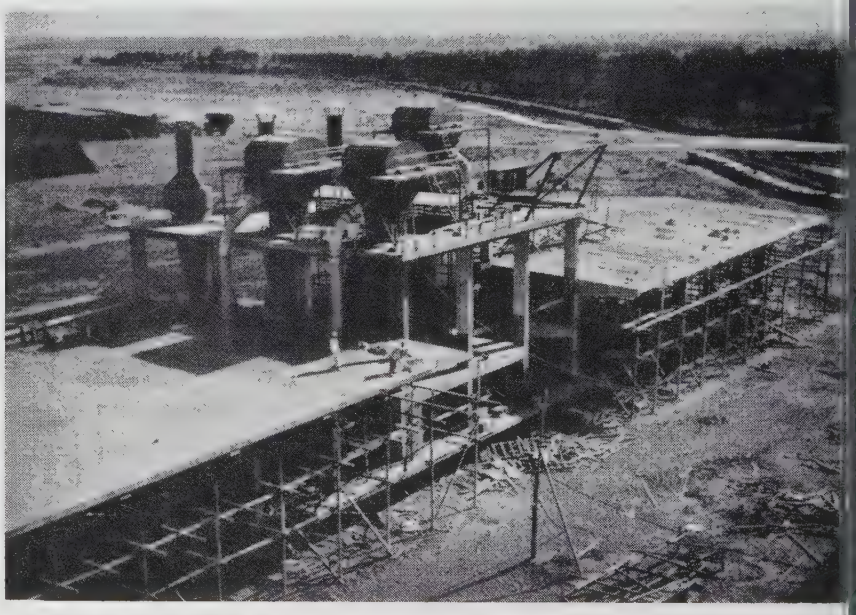
For more information about the efficient MgO pulping and recovery process, contact your nearest B&W sales office or write The Babcock & Wilcox Company, Boiler Division, Barberton, Ohio. E-302-1001

**Babcock & Wilcox**

## Pulp Mill in India

### 50 tons bleached pulp / day

out of bagasse (or grass) and bamboo (or wood) for the production of writing and printing paper.
All machines for the pulp mill as well as for the preparation of raw material, such as bagasse de-pithing and bamboo resp. woodchipping were supplied by us.
The mill started production some months ago.



## Pulp Mill in Egypt (U.A.R.)

### 80 tons bleached pulp / day

out of rice straw and reed for the production of writing and printing paper.
This mill is one of the few big pulp mills working on the base of rice straw.
The complete pulp and paper mill was erected by a German consortium, the leading member being Messrs. Fried. Krupp, Industriebau, production will be started in some weeks' time.

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# The Market Oriented Engineer

BENTON R. CANCELL

I APPROACH my assignment of presenting the keynote address at this conference with some trepidation—for the subject to which I address myself is one in which most engineers may feel they have but little interest. The subject is "The Market Oriented Engineer" and concerns the obligations and relationship of the engineer to the sales and marketing problems of his respective company.

Mr. Sawtell's letter asked people to come and study "the progress which the last year has given us." Admittedly the last year has seen major and significant engineering and technical advances, but in all too many instances, these advances have had but little relation to a well-conceived marketing philosophy—and being so unrelated, have often failed to achieve the economic and profit goals for which each company strives.

I propose to talk more about the obligations and opportunities which lie ahead of the engineer if we can aggressively overcome the wide gap which exists between engineering and marketing and if we can fill that vacuum with a high degree of teamwork, mutual confidence, and an understanding of the common problems between engineering and sales.

Despite the often-expressed fears of certain Federal Government Agencies that competition in the United States is lessening because of mergers and consolidations, the fact is that competition in the pulp and paper industry is aggressive and severe, both in domestic and foreign markets. Such competition has manifested itself in the rapid flow of improved products, new products, and new processes—aimed at expanding market opportunities, reducing costs, and expanding profits. The fact is, if one examines the financial record of the pulp and paper industry, that with very few exceptions, the trend of profits has been downward (almost in reverse proportion to new technical developments), both as a return on sales as well as on capital invested in the business.

Moreover, competition has been increasing between the pulp and paper industry and the products of other industries, notably plastics in its various forms. Just as dissolving pulp, paper, and paperboard made serious inroads into the consumption of silk, cotton, burlap, and lumber in the respective areas of textiles, bags, boxes, and other packaging, so we are now feeling the aggressive competition of synthetics in the pulp, paper, and paperboard industries. Fortunately, intelligent technology and engineering have found that in many instances paper, paperboard, and plastics complement each other and combinations of these materials have resulted in new products and new and expanded markets.

But competition in the pulp and paper industry is not a domestic affair only. Competition from foreign producers, both at home and in overseas markets, has now become a fact of urgent importance to American industry. The remarkable technical and economic development in Western Europe and Japan has changed the entire complex of world trade relationships between our industry and the producing industries of these various countries. The easy time that prevailed immediately after, and for some years following World War II, and the period immediately following the Korean War, when there was a great unfilled domestic and foreign demand for the products of the pulp and paper industry, has vanished. Barring a major war—which I for one do not believe will come—the easy domestic and foreign competitive situation will not recur—at least in the lifetime of most of us here.

To illustrate my point, in the last decade finished manufactured goods made up the bulk of American exports. But in the past seven years, exports of American goods have increased only 8% while world trade in such items has increased 40%. The balance of that increased trade is, of course, going to other industrialized nations. Significantly, during that same period our imports of finished manufactured goods has increased 250%.

True, American exports of pulp and paper have increased beyond the average of other American goods during the seven years referred to, but such exports have fallen far short of balancing the overcapacity which has existed in the pulp and paper industry and at the same time, new foreign productive capacity has been expanding rapidly.

The establishment of trading blocs, politically so necessary to world peace (and I refer to the European Common Market, the Outer Seven, and the probable establishment of a Latin American Common Market), pose a further challenge to the American pulp and paper industry for we no longer have a "corner" on technology and mass production methods. Nor has there been any real evidence of an awareness on the part of organized American labor of the responsibility and obligations which it bears to help keep American industry competitive in this shrinking world.

If, as individual companies, we are to meet this competition against other domestic producers of pulp and paper, of other domestic producers of substitute materials, and of foreign producers in both domestic and foreign markets, we must then be prepared to produce better goods at lower costs and to aggressively sell and service customers who may have a wide choice of suppliers and products from which to choose.

You may well be saying at this point "What has all this to do with me as an engineer? Is it not management's responsibility to take care of these matters?"

Do you remember the admittedly corny story of the man interviewing bricklayers on the job? One said he was working so that the family could eat; a second said he was carrying bricks up the ladder and putting

BENTON R. CANCELL, Executive Vice-President, St. Regis Paper Co., New York, N. Y.

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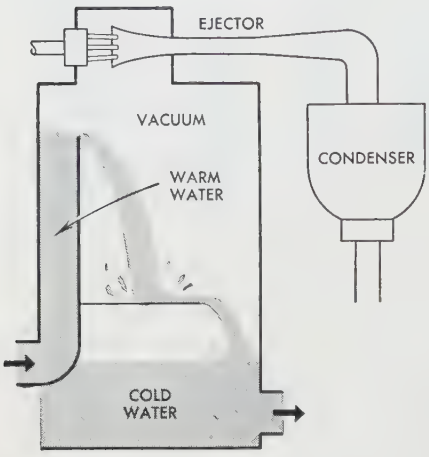


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them in place; a third said he was building a great cathedral. The third man had imagination and an understanding of his significance in the scheme of things—*an understanding of the end use of the work he was doing.*

No technical or engineering project should ever be undertaken as an end in itself. It must have some underlying purpose which may be one or a combination of:

1. Maintenance of the property for reasons of safety or maintenance of production.
2. Maintenance of quality products.
3. Improvement of quality of products.
4. Expansion of capacity, or
5. Reduction of costs.

In planning and executing any project, engineering and technology are not, and cannot be, an end unto themselves. Their place in the picture is as one cog in the corporate scheme of things and their ultimate goal must be to enable the sales effort to successfully serve the market place against the inroads of competition. Engineering planning which ignores these ends can be serious or even disastrous to a company—as is evidenced by the bodies on the historical battlefield of American corporate life.

Some years ago, a series of cartoons entitled "The Little Scouts" appeared in the *Saturday Evening Post.* One such cartoon made a great impression on me and illustrates my point. It showed one little boy scout dangling over a high cliff reaching into an eagle's nest for the eagle's eggs. Another scout was holding his feet. The angry mother eagle was swooping down with outstretched talons. The dangling scout was shouting pathetically to his friend "Maybe we should have found out if there was a market for eagle's eggs before we started this."

Let us take a look at sales for a moment. The successful salesman in our industry has changed completely. The glad hand, the entertainer, and the golf course devotee no longer gets the order unless he has something else to offer. In the sales departments of my own company we endeavor no longer to hire a young salesman unless he is prepared to spend many months as an in-plant trainee, learning at least the basic rudiments of manufacturing, technology, and engineering so he can understand how all of these functions contribute to a successful sales effort. Admittedly, we have a long way to go, for these programs should have been started many years ago.

Similarly, I hope our engineering people will gain a parallel understanding of the problems of sales and of their responsibility in helping the salesman meet the requirements of the market.

To sum up, may I say that the engineer who will move back far enough from the job to see it broadly, will see it take on more significance and meaning. He will then see more clearly the challenge, the obligations, and the opportunities which lie ahead.

He will then see more clearly the need for a closer working relationship between the engineer and people in the technical, manufacturing, and sales field (even though their basic philosophies and functions may vary widely).

He will then see that social contact with these other groups is not enough and that a singleness of purpose and common goal within the corporation is worth striving for.

He will then see that the final success of a project rests in the degree of acceptance and use of the product or process in the market place.

And finally, that a closer and well-coordinated working relationship between people in the engineering, technical, manufacturing, and sales departments is essential for corporate success, and perhaps survival in this modern era of *rapidly changing* technology, *increasing costs* and *aggressive competitian.*

These are the things I mean by the term "Market Oriented Engineer," and I hope to see a robust, market oriented TAPPI organization help our industry meet the competitive problems that lie ahead of all of us in these "slugging sixties."

Presented at the 16th Engineering Conference of the Technical Association of the Pulp and Paper Industry, held in Washington, D. C., Oct. 16–19, 1961.

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# Paper versus Plastic versus Paper and Plastic

BRAGE GOLDING

**The advantages and disadvantages of paper and plastic films and their combinations are cited. It is suggested that the paper industry must conduct more research on the components of wood if it is to hold and expand its position in the packaging field.**

I GREATLY appreciate the opportunity to talk to you this evening on a subject which is of great personal interest to me. My particular field of professional interest is that of plastics, or, more generally, polymers, but it so happens that I have had a number of years of industrial experience which were closely associated with the pulp and paper industry. As the director of research of an industrial finishes company, I had the opportunity to become well acquainted with a number of paper companies and, during that period of time, had the pleasurable experience of making many friends in the industry, several of whom are present tonight.

It seemed appropriate at this 16th Plastics-Paper Conference of TAPPI that I make some general comments concerning separately the fields of plastics and paper and then comment briefly on the progress and prospects of paper and plastic combinations, all as applied to the packaging industry. Following this, I shall doubtless make myself unpopular to some of you by addressing a few pointed remarks concerning research attitudes to your management and top technical personnel.

Let me begin by mentioning that there are a number of plastics which have been and are being used in large quantities by paper converters. Included among them are cellulose acetate, Pliofilm, polypropylene, polystyrene, polyesters, polyethylene, saran, and vinyl resins. There are many other polymers of interest, but those mentioned are probably the ones of greatest present importance. Most of these plastic materials have been known for some time and, in fact, most of them have been used for a number of years in the plastics industry. Excluding the use of polymers and resins as additives in the manufacture of paper (which is a large field in itself), all of the above mentioned materials are available commercially as films, whereby they compete directly with paper products. Whereas 20 or 25 years ago paper was the undisputed master in the packaging field, today plastic films compete not only with paper but among themselves for a very large share of the ever increasing packaging business. Since the costs of these wholly synthetic or modified natural products cover a rather wide range, bracketing the cost of many paper products, there must be reasons other than merely cost for their wide acceptance as packaging materials.

BRAGE GOLDING, Head, School of Chemical Engineering, Purdue University, Lafayette, Ind.

Let me approach this problem of paper versus plastics by first pointing out some of the advantages and disadvantages of paper as a packaging material. Obviously some of my comments will refer to an average condition, specific situations varying widely.

The use of paper has long preceded the use of plastics as a packaging material and paper continues to be used in very large volume as a packaging material, therefore there must be some real advantages in its continued use. Among these I would include its generally reasonable cost, its good flexibility, fair strength, its wide range of opacities, good printability, high insolubility, low toxicity, and good colorability. All of these qualities enables one to see readily why paper fulfills many of the major requirements for a good packaging material.

There are, on the other hand, a fairly impressive list of disadvantages which can be enumerated. Among them are the fact that the huge volume of production and type of equipment used in production make modification of paper during manufacture a rather difficult operation. In addition, paper lacks heat sealing characteristics, is flammable, has only fair aging properties, is very porous (thus has poor moisture and water vapor resistance), has little elasticity, has fair to poor optical transparency, has only fair electrical properties, because it is a nonwoven felt and is hydrophobic, has low wet strength, is relatively easy to tear, is fair to poor in bursting and impact strengths, has a relatively high density, and requires a high investment in the producing plant—not only in the buildings and equipment required but also in the peculiar requirements for plant location, such as the need for and availability of a very large volume source of water and, in many cases, the need for proper treatment of the effluent from the papermaking operations before the water can be returned to its source.

As a result of many of the disadvantages enumerated above, the addition, at some time or another during the manufacturing operations, of resins or other chemicals to upgrade paper products has been common practice. Typical examples would be the addition of certain polymeric substances to improve the wet strength of paper, the addition of certain inorganic chemicals to reduce the flammability, other purification steps or additives to retard aging, the addition of certain specific chemicals for plasticization, etc.

If one now turns to the plastic film field, one can also fairly readily enumerate a series of advantages and disadvantages. Among the advantages, generally speaking, are the better MVT, water, and chemical resistances; the unusually good flex properties; the easy heat sealability of many polymers; the greater impermeability; the ability to form very thin films; the greater optical clarity, generally excellent electrical properties, lower flammability, greater grease resistance and smoother surfaces of most plastic films; and

What an

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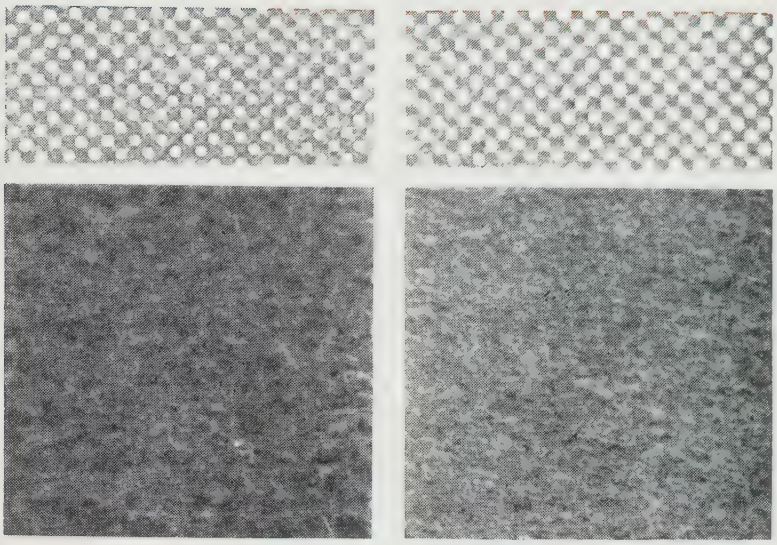

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in addition, the often easier fabrication because of the better solubility and thermoplastic properties of many polymers. As a result, they can usually be cast, extruded, calendered, vacuum molded, etc.—means of fabrication which are generally not available to the paper manufacturer.

There are, of course, disadvantages or drawbacks to the use of many polymers or plastics as packaging materials. Included would be the medium to high cost per pound of many of them (frequently offset, however, by their relatively lower densities), their fair to poor printability without special treatment, and their generally low use temperatures.

I think an important point to be made, one which does not often come to mind, is that because of the large number of polymers or synthetic resins available, compared to essentially one chemical base for paper, a much greater variety of special properties among the plastic films can be obtained. For example, one can start with different monomers, can combine them to form an almost endless number of co-polymers, and can vary the molecular weights, as well as the molecular weight distributions, of polymers over a wide range—and these variations will affect many of the physical properties of the final products. Paper, on the other hand, being essentially a nonwoven natural cellulosic mat, can be varied—at least according to present technology—in a relatively small number of ways. It is also noteworthy that paper behaves like a thermoset material although not truly one, while plastics are available both in thermoset and thermoplastic varieties, the latter being generally more useful in the packaging field. This thermoset property automatically imposes a rather severe limitation on some of the useful properties which paper might otherwise have.

I do not mean, however, to leave the impression that all papers are alike. Certainly this is not true. In fact, I am sometimes amazed at the variety of, at least superficially, different products which can be obtained from the same natural cellulosic base. My comments made earlier, however, must still hold.

Now, turning to the field of combination of paper and plastic materials, it is obvious that, since there is a market in these combinations, there must be good reasons for their use. Since the very process of combining paper and plastic involves manpower and equipment, the mere act of producing such a product must yield one which is inherently more expensive. That is, if a paper or plastic material alone exhibited all of the properties desired, then no composite would compete unless one of the components lowered the cost without detracting from the good properties of the other material. Alternatively, one of the components must contribute to lower cost in the final product in spite of the additional operations required to form the combination or must contribute some special property not obtainable from the other component alone. Again, I am limiting my comments to combinations of plastics with paper which are formed after the paper is made—by such processes as extruding, laminating, coating, etc.

Typical examples which come readily to mind would include the addition of a polyethylene film to a paper backing to improve the moisture vapor transmission resistance, scuff resistance, or abrasion resistance of the paper while at the same time giving to the plastic film the necessary rigidity which paperboard would impart; the application of a lacquer coating to a porous paper to improve its gloss, grease resistance, or moisture penetration while the paper functions as a substrate to improve rigidity, provide a surface for printing, and at the same time reduce the cost as compared to the much thicker plastic film necessary if it were used alone.

Although I have tried to think of many different reasons why paper is useful in paper-plastic combinations, it seems that I come back to essentially one principal one -that it provides more rigidity at lower cost. It is hard for me to conceive, therefore, of a very bright future for the paper-plastic laminated products so far as future rapid growth and acceptance are concerned. Unless the paper component can also contribute better properties, physically and/or chemically, than it does now, a real expansion in its use in combinations appears to me to be limited.

Although the packaging industry is only one of several which the paper and plastics industries supply, it is nevertheless a very large consumer of these materials. Whether the paper industry manages to hold its own in this very competitive field or whether it will gradually lose out in even larger measure to the plastics industry depends on many factors.

This brings me to my next point concerning one of these factors. It is likely that if paper manufacturers continue—as many, but not all, appear to have done in the past—to compete with one another by merely trying to reduce slightly the cost of the same products they have made for many years, then their chances for real progress in paper technology and new uses for paper appear limited.

My observations of real research in the paper industry—one of the largest in the world—have led me to believe that the industry has a wonderful future—because it has done so little so far! I have the impression that if one were to compare the ratio of gross sales to expenditures for real research, or the number of qualified research personnel to those in other technical positions, or even the attitude of top management toward fundamental research in many paper companies to that of other chemical industries, he would be unfavorably impressed.

The Institute of Paper Chemistry, a few universities and a few progressive paper companies appear to me to be the only organizations really concerned with fundamental progress, and many of the paper companies which are doing worth-while research have become involved on a serious scale only quite recently. Too many technically qualified men in paper laboratories appear to be doing routine mechanical evaluation of products rather than engaging in real research.

In my many contacts with both technical and management personnel, I have received the impression that some paper companies are extremely conservative and take unduly long to consider a change to a better method of manufacture or to use a better intermediate product. It often appears to take *years* for some managements to make a decision to experiment on a change. While it is realized that the nature of the industry, the size of production, and the massiveness of the whole process makes for less flexibility, it would seem that much more rapid progress should be possible.

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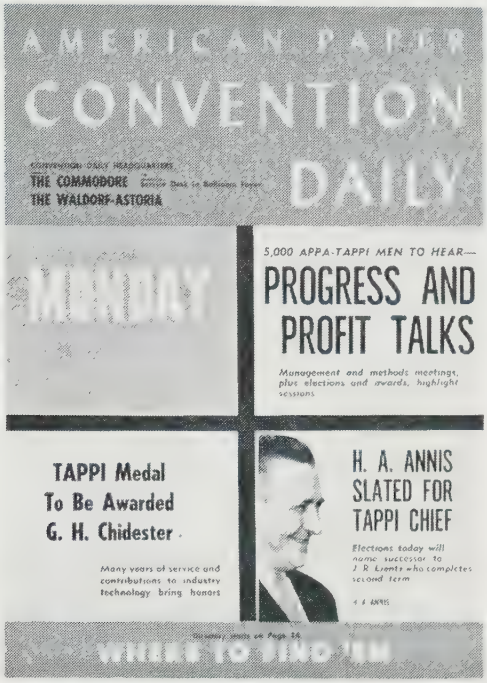
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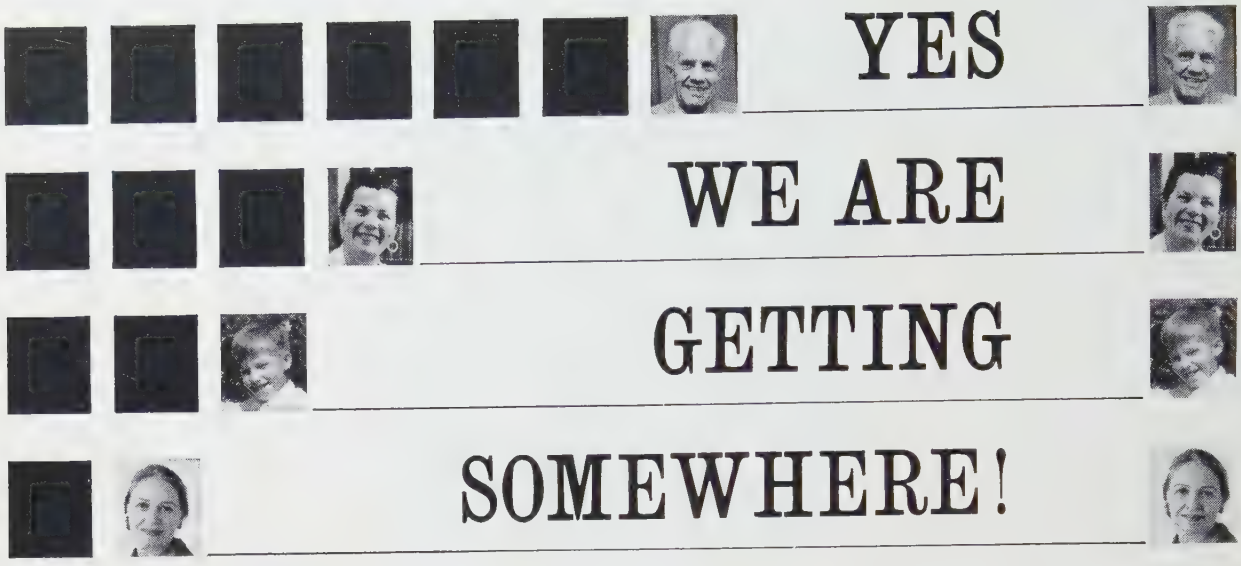

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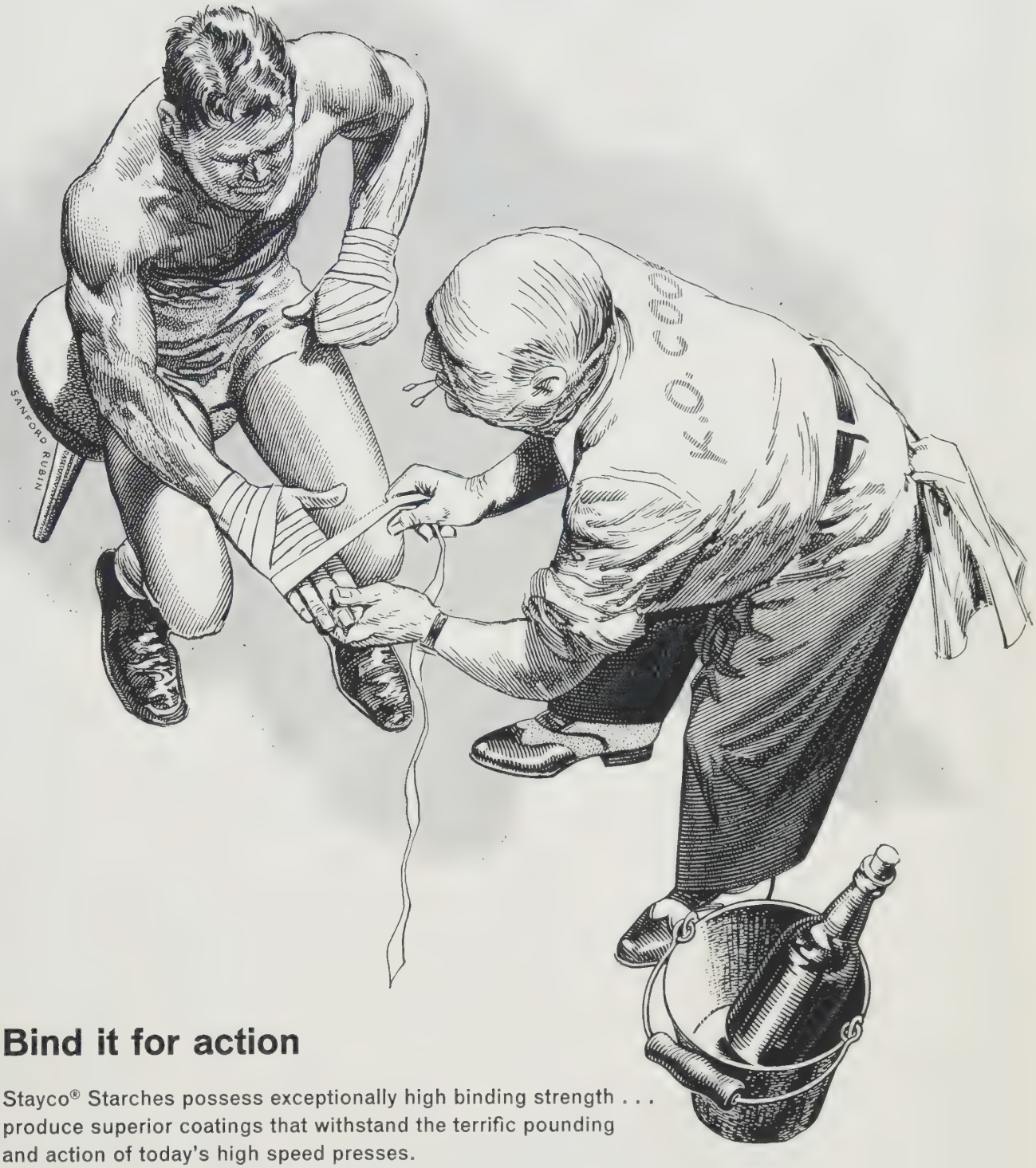


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On the technical side, I wonder how many paper companies assiduously solicit the physical and organic chemists and the chemical and mechanical engineering graduates from universities, as compared to the other chemical industries and, once having employed some highly trained technical personnel, use them for *creative* work? How many of these companies have more chemists and chemical engineers knowledgeable in the plastics and polymer field than staff experienced in the mechanical and analytical fields? The impression persists that at many paper meetings—including some of those of TAPPI—a great proportion of time is spent in discussions of testing and evaluating procedures (certainly necessary) but less time seems to be devoted to research on new and improved products and chemical processes. You are in the chemical and, more specifically, the polymer industry, yet the emphasis seems often to be more on the mechanical problems than the chemical ones. A slight improvement in a mechanical feature often is far less significant than an entirely new or upgraded product.

Consider, for example, some of the facts about cellulose, the chief material with which you are dealing in all paper and converting operations. Cellulose is the major constituent of plants and is the most abundant and widely used organic chemical in the world. Billions of tons of it are created annually by photosynthesis. This polymer is the fibrous component of vegetable tissues which gives them coherence and form. It occurs in combination with noncellulosic carbohydrates, their acidic oxidation products, and with the lignins. Although cellulose is a universal component of the plant world, two specific sources account for the vast majority of uses: wood and cotton. For various reasons, the paper industry is concerned chiefly with the cellulose obtained from wood. Although a large quantity of cellulose is chemically converted into derivatives which are useful in the textile, film, plastics, and coatings industries, the material with which we are here concerned is cellulose more or less in its native state.

It seems to me somewhat surprising that, with such a huge supply available, more has not been done from the research point of view to determine whether, by a slight change in its properties, either by physical or chemical means or both, superior or unusual products might not be obtained. There are many synthetic organic substances, used on a far smaller scale, which have been considerably more intensively investigated. The argument that cellulose as obtained naturally is a complex mixture of many things and that it varies according to source is well founded and does indeed complicate the research effort. However, modern instrumental methods of analysis as well as the development of many new chemical techniques in recent years should provide some of the keys to unlocking the fine structure of cellulose and help us to understand how various modifications may change its properties.

As you all know, pure cellulose is a linear polymer composed of thousands of anhydroglucose units linked at the one and four positions through $\beta$-glucosidic bonds. The anhydroglucose units consist of pairs of rings connected by oxygen atoms, each ring containing an oxygen atom and also three hydroxyl groups, two of them secondary and one primary. These are indeed fertile molecules for the study of possible chemical and physical variations.

One of the problems associated with investigation and modification of cellulose is attributable to the fact that cumulative hydrogen bonding along the molecular chains has produced a polymer which shows appreciable crystallinity (that is, some degree of microregularity), low solubility in almost all common solvents, and high infusibility. Naturally, these properties are of great value to plants, but these same properties detract from some of the uses to which we wish to put this material.

While I am not and do not claim to be expert in the field of cellulose chemistry, it would seem that more detailed investigations could be made of the effects, for example, of variations in molecular weight and molecular weight distribution with respect to the final physical properties obtained in a finished paper product. While we cannot expect to increase the molecular weight of natural cellulose, except perhaps by cross-linking it, certainly it can be degraded by various means to obtain a whole range of lower molecular weight products.

We also know it is possible, under what might be called fairly drastic conditions, to solubilize native cellulose. In all solubilizing procedures, advantage is taken of the fact that the intermolecular forces among the cellulose chains must be lessened or destroyed

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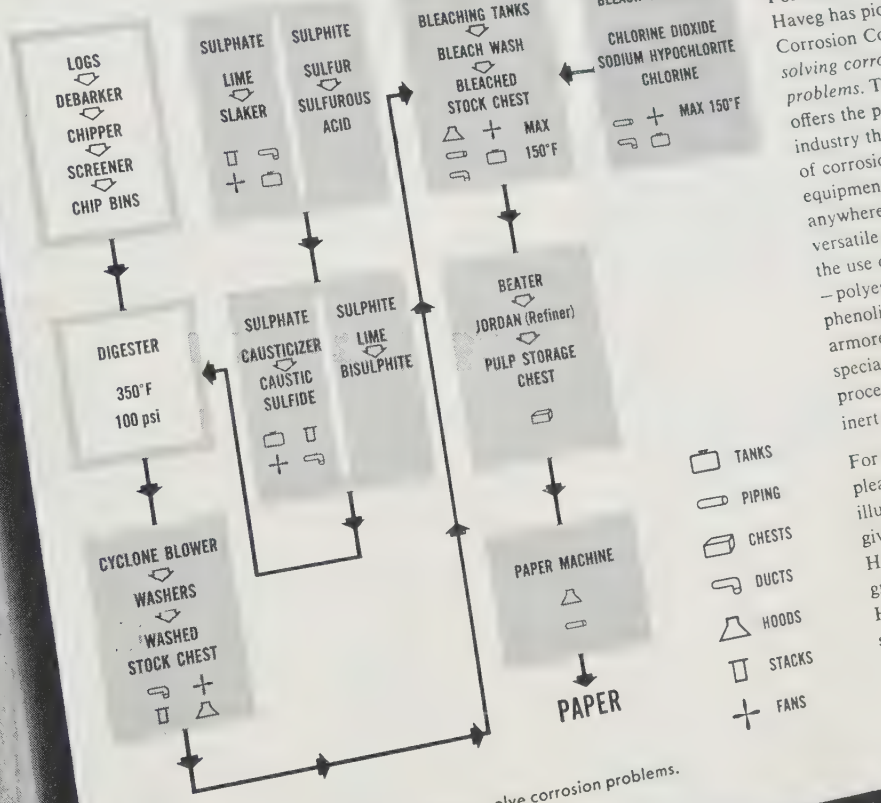


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by some means or another. Chemical modification, such as by the use of sulfuric, acetic, and nitric acids, to destroy regularity or form derivatives which replace some of the hydroxyl groups with larger or less strongly hydrogen-bonded groups, is well known. Chemical conversion to an ionized and more solubilized form, such as is used in the viscose and cuprammonium rayon processes, is likewise well known. Perhaps it would be possible to merely swell the cellulose fibers by one of the above techniques or by a new technique and then, without dissolving the fibers, examine the products for their utility in papermaking operations. It also seems possible that clever chemists may find other ways to decrease the regularity of the chains and the degree of crystallinity of the gross product and thus change many of the physical characteristics of native cellulose.

I do not even know whether alkali or soda cellulose, which is easily regenerated to form a physically modified cellulose, as is done to make mercerized cotton, has ever been extensively investigated as a raw material for paper products. Naturally, the first thought is: "Look what this will do to the cost!" and I must agree that the products will doubtless not compete with many of the ordinary uses for paper. However, many products have been made in the past which were more expensive—indeed, must inherently be more expensive—than their precursors, and these have sold well because they had properties not available in their parent compounds.

It would clearly be foolish to suggest that the greater proportion of paper be made by a new and more expensive process, even if it did have better properties, because there are countless uses for paper for which the present product is satisfactory. Certainly, however, with such an abundance of raw material and with the knowledge and ingenuity available today, *if it is used*, it should be possible to discover new and very useful variations of cellulose.

To carry my argument a step further, the other chief component of wood is lignin, which serves as the binder for cellulose. Millions and millions of tons of lignin are available annually, much of it a by-product of your own industry. Although many attempts have been made to utilize this inexpensive, thermoset polymer, most of it is disposed of as waste. A few paper companies have done some research into the elucidation of the structure and composition of lignin and have also found some selective uses for it, such as a control agent of viscosity in oil well drilling muds, as a dispersant, as a tanning agent, as an extender or replacement for a portion of certain resins in plastics manufacture, as a raw material for the manufacture of synthetic vanillin, and as a reinforcing agent for various elastomers, but so far as research and development on really large scale uses are concerned, the surface has only been scratched. With so much raw material available at such low prices and with the available supply being constantly replenished, much more intensive and extensive work appears indicated. In consideration of the factors of availability, replenishability, and cost, it may well happen that some day, in the not-too-distant future, such by-products may well be of greater value to the producer than paper—provided management follows the lead of many other chemical companies and encourages and supports active investigation of other avenues of profitability not necessarily associated with the principal product being manufactured.

One cannot fail to think of the meat packing industry in this connection. Years ago, profitability and future growth along traditional lines appeared extremely limited. Therefore many big packers wisely undertook a strong research program designed to discover useful and profitable chemicals from by-products. It is now well known that profits in this industry come almost exclusively from its chemical by-products—a situation which benefits both the meat packing industry and the public, since it has enabled these companies to maintain their financial health while continuing to process and produce their meat products at reasonable prices.

Let me summarize by reminding you again that you are dealing with essentially only one raw material—that of crude cellulose. In order to improve your products or produce radically new ones, it is axiomatic that you must have a very specific and detailed knowledge of your raw material and of ways to modify it so as to improve its deficiencies.

In order to accomplish these goals, your management and top technical staffs must be research oriented and be willing to gamble on money spent now for a greater return later. Great improvement in this attitude has occurred in the paper industry since World War II but more is needed. Technical conferences, chemically oriented, such as this one, are a step in the right direction.

Presented at the 16th Plastics-Paper Conference of the Technical Association of the Pulp and Paper Industry, held at French Lick, Ind., Oct. 9, 1961.

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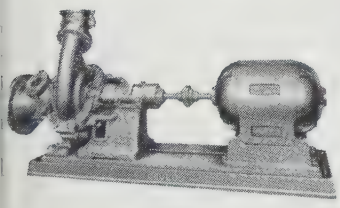
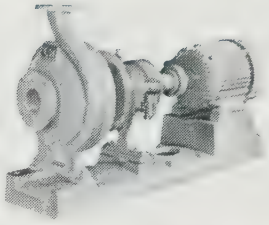
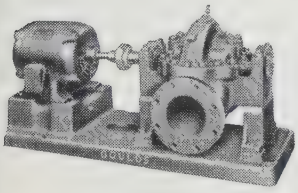


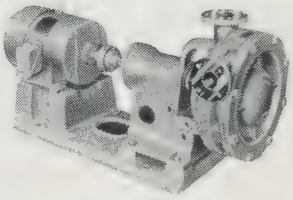
**Model 3135—STOCK PUMP, single-stage paper stock pump.** Centerline discharge and self-venting casing provide steady flow without binding. Non-clogging impeller with back wall ejector vanes. Twelve sizes, capacities to 6400 GPM, heads to 300 ft. Diagonally split casing saves time and effort removing rotating element without disturbing pipe connections. Ask for Bulletin 723.1. **Uses:** Paper stocks of all kinds, blowpit, bleach, broke.



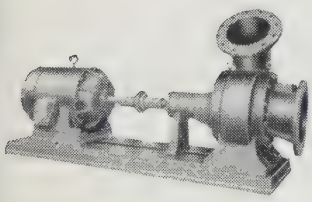
**Model 3195 — CHEMICAL PUMP.** 11 pump sizes with same overall length, 23 in. Capacities to 775 GPM, heads to 300 ft. TDH. Working pressures to 275 PSIG. Maximum interchangeability of parts and dimensional interchangeability of models give you greatest flexibility and coverage. Standard materials: ductile iron, 316 stainless steel, Gould-A-Loy 20. Handles liquids to 500° F. Ask for Bulletin 725.1. **Uses:** Liquor recovery, bleach liquid, size-and-alum.



**Model 3405 — GENERAL SERVICE PUMP, single-stage, double suction pump.** All sizes and pump combinations based on only 3 shaft and bearing assemblies. Mechanical seals or stuffing boxes. Rotation can be changed in field. 35 sizes, capacities to 6400 GPM, heads to 525 ft. Horizontally split casing for easy inspection, maintenance. Ask for Bulletin 721.6. **Uses:** Mill water, filtrate, fan pump, white water, centri-cleaner.

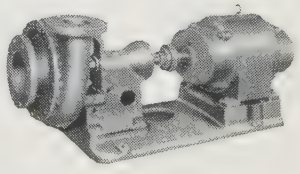


**Model 3708—GOULDS-PFAUDLER GLASSED PUMP.** Revolutionary new pump designed for handling corrosive liquids. All parts in contact with liquid are glass—fused to metal. Handles all acids and alkalies to pH 12. Four sizes, with capacities to 700 GPM, heads to 140 ft. Ask for Bulletin 725.2. **Uses:** Bleaching, other corrosives.



**Model 3138-9—STOCK PUMP, single-stage end suction pumps.** Vertically split, with discharge nozzle at 45°. Can be furnished in right- or left-hand rotation. Back pull-out design permits entire rotating element to be removed from casing without disturbing pipe connections. Externally adjustable impeller clearance. Capacities to 9500 GPM, heads up to 220 ft. Ask for Bulletin 725.5. **Uses:** Slurries or tailings, waste waters, digester circulating, bleach liquor recovery, stock handling, filtrate, bleaching, broke.

**Model 3115—STOCK PUMP, vertical construction. Vertical centrifugal stock pump.** Large passages allow free flow of heavy stock without air binding. Special warped vane, heavy-duty non-clogging impeller. Axial adjustment of rotating element for impeller clearance. Rotating element can be removed without disturbing pipe connections. Ask for Bulletin 723.2. **Uses:** Stock, broke.



**Model 3189 — STOCK PUMP, small capacity.** Heavy-duty centrifugal pump available in 13 sizes with capacities up to 3000 GPM and heads to 180 ft. at 1750 RPM. Design accommodates wide interchangeability of parts. Hydraulic design provides high operating efficiency with low power consumption. Wide openings give ready access to gland and stuffing box. Ask for Bulletin 720.4. **Uses:** Filtrate, white water, size-and-alum, broke, and centri-cleaner.

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**Sump.** For pit depths to 20 ft. in 6 inch increments. Both wet and dry pit types available in capacities up to 3180 GPM, heads to 190 ft. Single and duplex units. Full automatic control. Modern design using standard parts permits quick shipment at most economical cost. Bulletin 726.2.

**Process.** Especially designed for handling corrosive liquids in the chemical process and allied industries. Normally supplied in 316 stainless steel constructions but materials to suit user's requirements can be furnished on application.

Capacities to 720 GPM. Heads to 190 ft. For heavy-duty service under most severe applications. Bulletin 727.1.

**GOULDS PUMPS**

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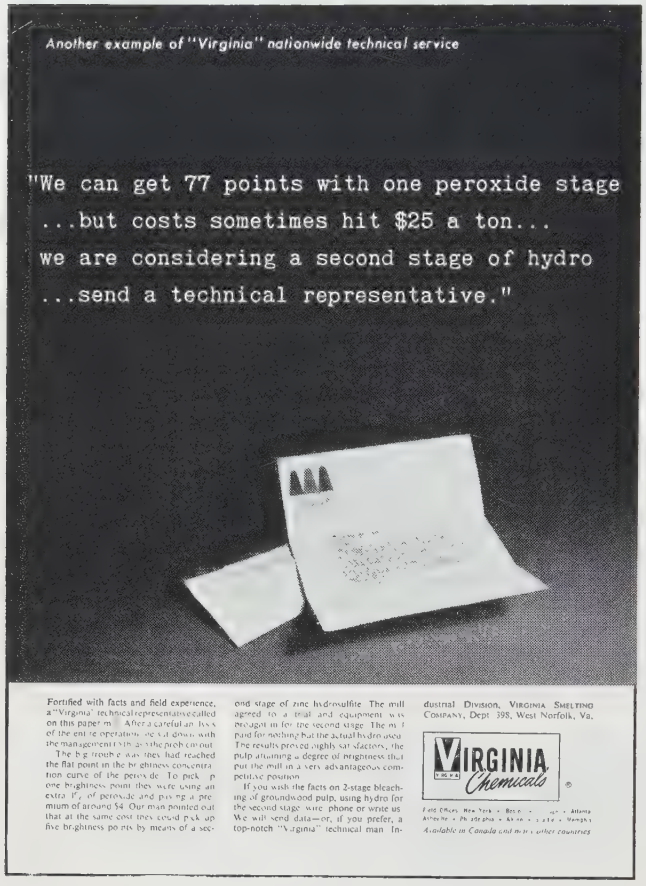
# LEADING MANUFACTURERS KNOW THE RESULTS


Another example of "Virginia" nationwide technical service
"We can get 77 points with one peroxide stage
...but costs sometimes hit $25 a ton...
we are considering a second stage of hydro
...send a technical representative."
Fortified with facts and field experience, a "Virginia" technical representative called on this paper m[ill]. After a careful an[alysis] of the entire operation, he sat down with the management in the pick[up]
The big trouble was they had reached the flat point in the brightness concentration curve of the peroxide. To pick up one brightness point they were using an extra 1% of peroxide and paying a premium of around $4. Our man pointed out that at the same cost they could pick up five brightness points by means of a second stage of zinc hydrosulfite. The mill agreed to a trial and equipment was arranged in for the second stage. The mill paid for nothing but the actual hydro used. The results proved sights set sky-high, the pulp attaining a degree of brightness that put the mill in a very advantageous competitive position.
If you wish the facts on 2-stage bleaching of groundwood pulp, using hydro for the second stage, write, phone or write us. We will send data—or, if you prefer, a top-notch "Virginia" technical man. Industrial Division, Virginia Smelting Company, Dept. 398, West Norfolk, Va.
VIRGINIA Chemicals
Field Offices: New York • Boston • Chicago • Atlanta
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"We offer free mill trials to show the fine results and low cost of groundwood pulp one-stage hydro bleaching–the only cost to the mill is for the hydro"
George V. Rittenhouse (left) and Robert W. Barton
Technical representative Bob Barton and salesman George Rittenhouse, both of Virginia Smelting Company, answer some questions regarding the efficiency of bleaching groundwood pulp with one stage of hydro.
Q Do zinc and sodium hydrosulfites provide the most economical method of one-stage pulp bleaching?
Mr. Barton—If the mill is going for anything between 2 and 11 brightness points, bleaching groundwood pulp with hydro is virtually always the cheapest method. It is easier to apply—there are no control tests involved and the chemical cost is much lower.
Q Is it difficult to set up a trial run for a mill that might want it?
Mr. Barton—Not usually. Frequently much of the equipment we need is available in the mill. We go in with our lab equipment and make tests, checking the pH we're going to deal with, and possibly deciding on the maximum temperature possible. We give all the facts to the mill—and if they want the trial, we set it up. Often the mill has a pulp storage chest. A few changes and we can use it as a bleaching tank. We'll bring in feeders, attach hoses, and start the trial.
Mr. Rittenhouse—Our technical men stay there until the setup is running perfectly and they make sure the operators know everything they need to. You'd be surprised at what we run into sometimes, even in mills that have been bleaching regularly with hydro. They take the powdered hydro and dump it in on top of the stock. Of course, hydro breaks down fast when it is exposed to air, and they wind up with reaction times too short—temperature not showing the proper effect—and poor brightness response at the end. I might add that faulty mixing like this can cause a corrosion problem, too—a problem that does not arise if proper air-free mixing is standard procedure.
Mr. Barton—A good point, George. This is the chief reason why some mills are under the mistaken impression that hydro always causes a corrosion problem. If hydro is handled properly there's no corrosion problem.
Q Isn't a mill trial a pretty extensive service at no charge?
Mr. Rittenhouse—Yes, I suppose so—but I consider it the most effective way of showing a mill the economy and the good results obtainable with hydro. After all, mill trial data offer the most believable evidence of what I say. And production doesn't have to be stopped—we can almost always set up the mill trial without affecting it.
Q What are the advantages to the mill in using a "Virginia" hydro?
Mr. Barton—There are several. One is the consistently uniform quality and purity of our hydro, either the zinc or sodium. It's a very clean, free-flowing powder—made, packed and shipped as moisture-free as modern manufacturing methods can make it.
Mr. Rittenhouse—And of course our technical service goes with it. A mill can always call for assistance and get a "Virginia" technical man. It's the accepted thing with our customers.
• • •
FOR A FREE MILL TRIAL
—just drop us a line, phone or wire, and a representative will call on you at your convenience
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# HYDRO BLEACHING—
# WHETHER 1-STAGE OR 2-STAGE—
# GIVES REQUIRED BRIGHTNESS AT LEAST COST

Men in the paper industry are noted for basing their decisions on facts . . . and anyone who has examined all the facts knows that hydrosulfite, whether used as a single-stage bleaching agent or the second stage in a 2-stage bleaching process, produces mechanical pulp at *any* brightness level more economically than any other method.

In single-stage bleaching to standard brightness levels hydro will give you more brightness, pound for pound, than any other known method. And at lower chemical cost. The reason is that hydro is a powerful reducing agent, particularly effective at low concentrations. Oxidizing bleaches require higher and more costly chemical concentration for comparable results.

For super brightness gains, 2-stage bleaching is a must, because in a single-stage oxidizing bleach the brightness reaches a flat point below the maximum attainable for the pulp. A second stage of hydro gives you those critical extra points—and more—at the lowest possible cost per point in the finished sheet.

So before you decide, get all the facts from "Virginia" . . . the industry's leader in groundwood bleaching technology. Call or wire us—we will send you complete data. Or, if you prefer, we will have a technical service engineer discuss your problem in detail.

Chemical Division, Dept. 398
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# PILOT COATER

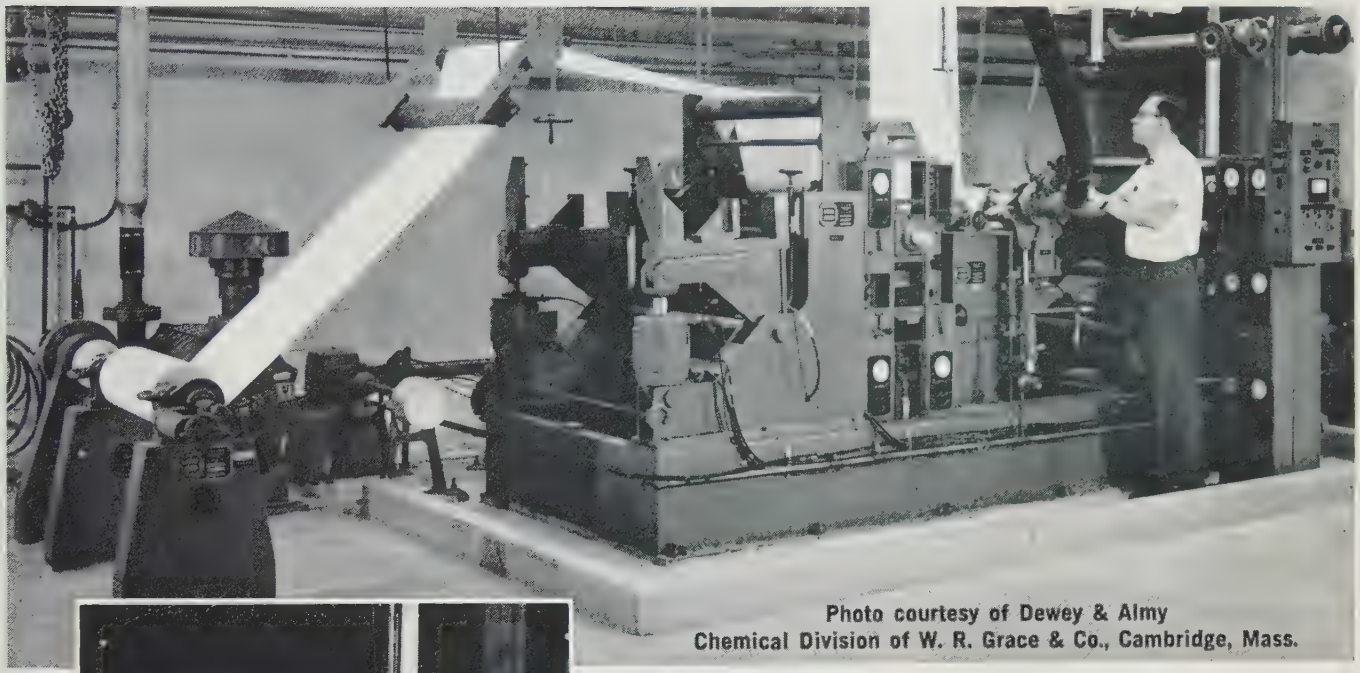
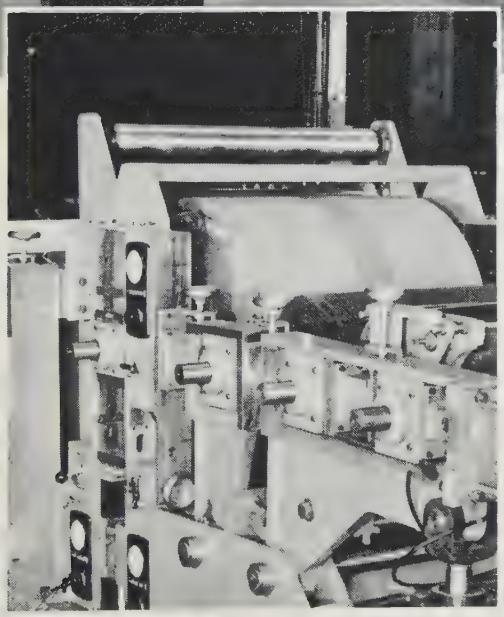


Photo courtesy of Dewey & Almy
Chemical Division of W. R. Grace & Co., Cambridge, Mass.



Close-up of transfer roll.

## *"Most Versatile and Accurate" Says DEWEY AND ALMY*

A coater to simulate production conditions with provisions for utilizing universal coating methods was required by Dewey & Almy Chemical Division of W. R. Grace & Co., supplier of latex paper coatings.

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The versatile five roll transfer coating head is easily converted to Air Doctor, Flexiblade®, horizontal and vertical size press methods. Speed goes to 2000 fpm.

Dewey & Almy use their Black-Clawson coater for: 1. Research and Development of new products; 2. Customer Service; 3. Sales Development; and 4. Advancement of paper coating technology.

Talk to a Dilts Division coating specialist and learn more about the Black-Clawson pilot coaters.

**DILTS DIVISION**
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***CONVERT WITH CONFIDENCE***

**BLACK-CLAWSON**

# Coating of Paper in Germany

M. JUDT

**In a short historical survey data on machine speeds and machine widths, predrying temperatures, and daily tonnages at the coating mills of Heidenau, Saxony, and Hillegossen, Westphalia, are given, showing the development of off-the-machine coating in the past 30 years. Raw stock properties and their influence on coater performance, 120-in. wide, are quoted. After presenting comparative data on coated papers made in the United States and in Germany some differences in quality and approach that have developed are discussed. On-the-machine and off-the-machine coated qualities are compared and the future of these techniques and their products in Germany is considered.**

At this conference, all have the same dream of producing or helping to produce, cheaply and quickly, a faultlessly beautiful coated paper or board that will satisfy our customers, and so the author thought when the chairman of the Program Committee asked him to give a paper, that perhaps his company's efforts in this direction in the past and its hopes for the future could be of interest.

The author's company is the Feldmühle Paper and Pulp Mills Corp., and it is the biggest paper and board producer in Germany and the coating mill at Hillegossen is one of the largest off-the-machine coating mills in Europe.

To start the author would like to give a short historical survey of the development of coating of paper in the past thirty years. By doing so you can become acquainted with the background situation, the traditions, and the opportunities of coating in Germany. The following story with some variations could be the story of other coating mills in Germany.

## COATING OF PAPER AT HEIDENAU, SAXONY

In 1935, the coating mill of Krause & Baumann was merged with the Feldmühle. This mill, founded at 1867, was at that time renowned for the fine quality of its coated art papers and boards and its manufacture of playing cards. By this merger the production facilities of coated printing papers were added to the manufacture of chemical wood pulp in three pulp mills and of uncoated printing papers such as newsprint and rotogravure and packaging papers and boards in seven other mills.

In this coating mill at Heidenau, Saxony, seven single cylinder brush coaters with festoon drying (Fig. 1) and nine two side vertical brush coaters with tower predrying and festoon drying (Fig. 2) produced up to 85 tons of coated paper per day, to the author's knowledge the highest production of coated paper per mill in Europe at this time of the century.

In 1938 the maximum tonnage of the widest single coater, 41 in. trim width, was 6.2 tons per day, with speeds up to 200 f.p.m.; of the two side coater, 51 in. trim width, 6 tons per day with speeds up to 165 f.p.m.

In 1940 a tenth two side coater with horizontal tunnel drying was installed. This machine had a width of 70 in., the maximum speed of coating ever obtained was 220 f.p.m.

Until the war casein was the primary adhesive. In 1940 enzyme conversion of starch was introduced, and in 1943 the first successful trials with acrylic emulsions as binders were made.

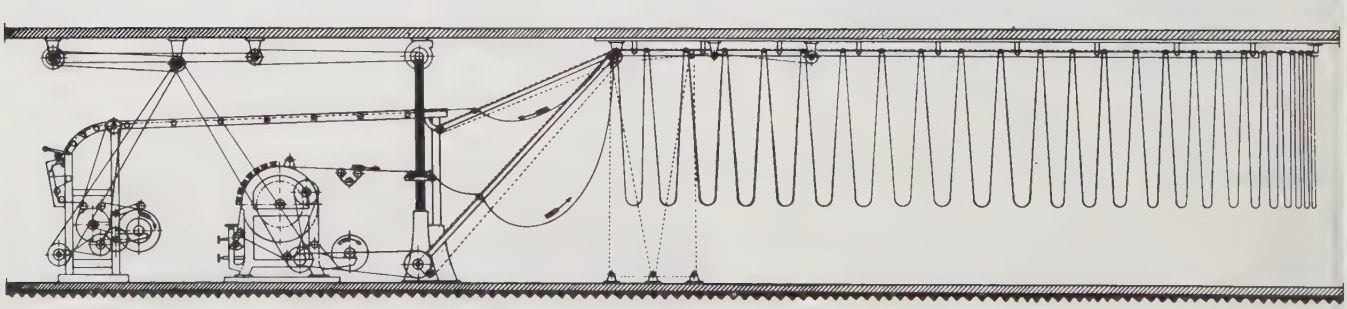
Due to the fact that the mill was able to supply a substantial amount of binding material from its own source the production of coated paper did not come to a stop during the war. This source was found in the effluents of a viscose mill nearby. By centrifuging and collecting polysaccharides from this water a binder material was procured which gave a satisfactory coated sheet. The binder ratio of the adhesive was: 17 parts of casein sufficed to give an acceptable grade; 20–24 parts of starch did the job too, but 40 parts of polysaccharides were needed to give similar results. To the author's knowledge this interesting source of adhesives has never been tapped again.

After the Second World War this mill was dismantled and all its machinery, including 17 coating machines, were shipped to the Soviet Union. One wonders whether these machines now produce coated paper there.

## COATING OF PAPER AT HILLEGOSSEN, WESTFALIA

After the headquarters of the company was moved

M. Judt, Assistant General Manager, Feldmülde Paper & Pulp Mills, Corp., Hillegossen, West Germany.



Single cylinder brush coater with festoon drying (1930)

*Fig. 1*

Everything can be improved. The progress of the papermaking industry is proof that constant improvement of techniques is the reason for today's better paper and constantly increasing production.

It's the same with felts. The result of applying proved research, refined production and design techniques, and up-dating treatments is ever-better felts . . . and this means **ever-improving paper production.**

Making felts is a complex business. It must be progressive!

Albany Felt Company is able to draw on the world's outstanding researchers, chemists and felt processors and thereby keeps its techniques up-to-the-second. As an international organization, engaged in felt manufacture in the UNITED STATES, CANADA, SWEDEN AND MEXICO, Albany Felt Company both learns and teaches in a free exchange of technical information and felt-performance experience.

Sharing information with talented feltmakers as well as talented papermakers around the world means felts that get better and better. **That means even better paper production.**

AF

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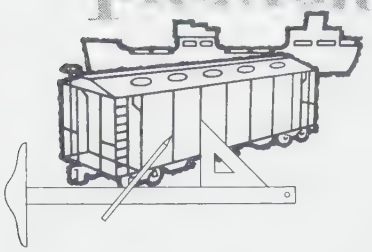

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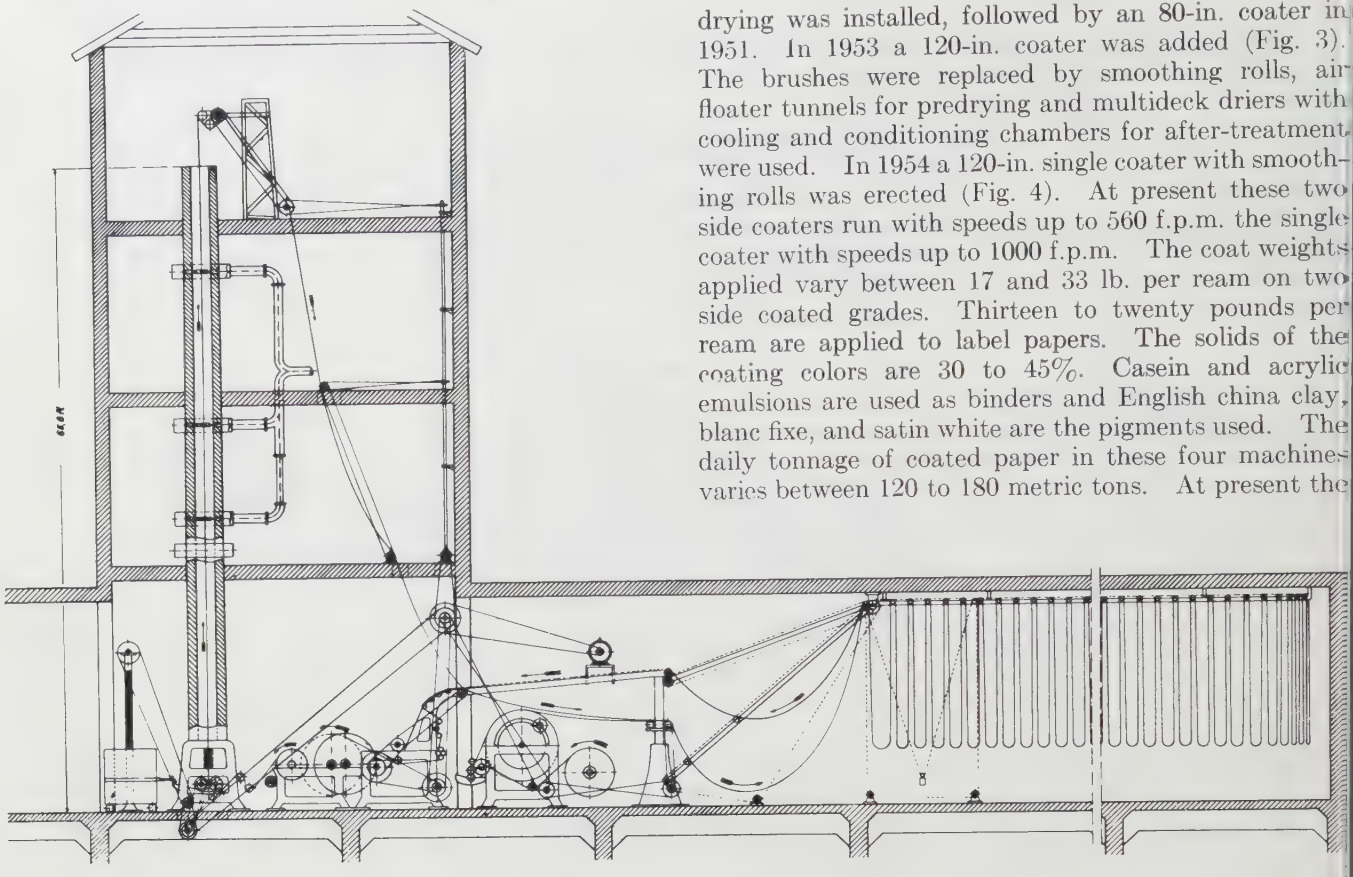
*100°F., 90% RH **Look at these properties . . . and compare.**

| PROPERTY | TUFFIN 10 | TUFFIN 20 | TUFFIN 30 | PARAFFIN WAX A | PARAFFIN WAX B |
|---|---|---|---|---|---|
| Melting Point, D87 | 132 | 150 | 143 | 131 | 143 |
| Viscosity, cs @ 210°F. | 79 | 125 | 2300 | 3.4 | 4.2 |
| @ 250°F. | 50 | 74 | 1300 | — | — |
| Color | +25 | +25 | +25 | +30 | +30 |
| Blocking Temperature, °F. | 105 | 132 | 120 | 100 | 117 |
| WVTR-g/100 sq. in./24 hrs.* Flat | 0.36 | 0.43 | 0.5 | 0.58 | 0.2 |
| Creased | 1.19 | 0.86 | 0.5 | 3.53 | 4.2 |
| Seal Strength, g/in. Sulfite to sulfite | 145 | 64 | Tear Seal | 15 | 17 |
| Glassine to glassine | 25 | 35 | 40 | 4 | 1.7 |
| Sulfite to aluminum foil | 205 | 56 | Tear Seal | 12 | 20 |

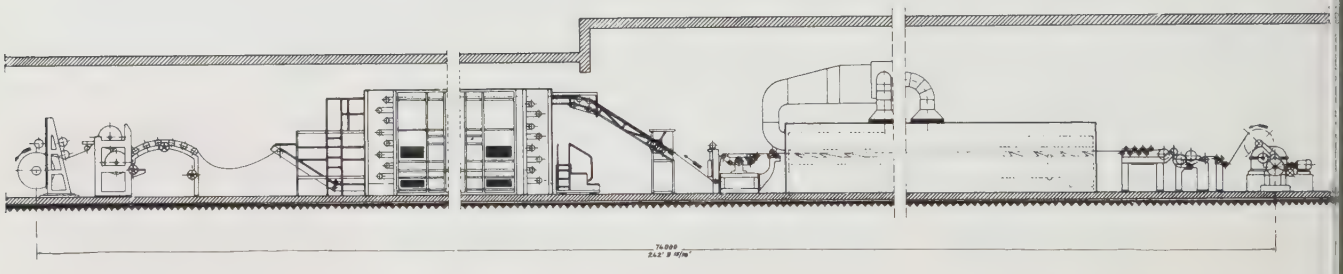
from Berlin to Western Germany in 1945 all the pulp mills and two paper mills and the only coating mill had been lost. A new beginning had to be made. Therefore in 1948 the board of directors decided to start the production of coated papers from rock bottom in an old newsprint mill at Hillegossen, Westfalia.

In 1949 a 40-in. brush coater with horizontal tunnel drying was installed, followed by an 80-in. coater in 1951. In 1953 a 120-in. coater was added (Fig. 3). The brushes were replaced by smoothing rolls, air-floater tunnels for predrying and multideck driers with cooling and conditioning chambers for after-treatment were used. In 1954 a 120-in. single coater with smoothing rolls was erected (Fig. 4). At present these two side coaters run with speeds up to 560 f.p.m. the single coater with speeds up to 1000 f.p.m. The coat weights applied vary between 17 and 33 lb. per ream on two side coated grades. Thirteen to twenty pounds per ream are applied to label papers. The solids of the coating colors are 30 to 45%. Casein and acrylic emulsions are used as binders and English china clay, blanc fixe, and satin white are the pigments used. The daily tonnage of coated paper in these four machines varies between 120 to 180 metric tons. At present the



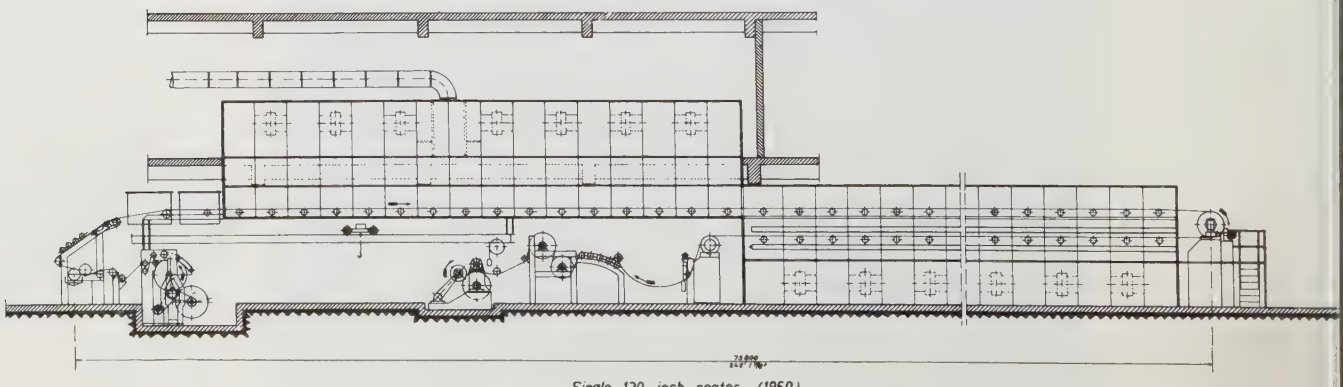
Two side vertical brush coater (1930)

*Fig. 2*



Two side 120-inch-coater (1960)

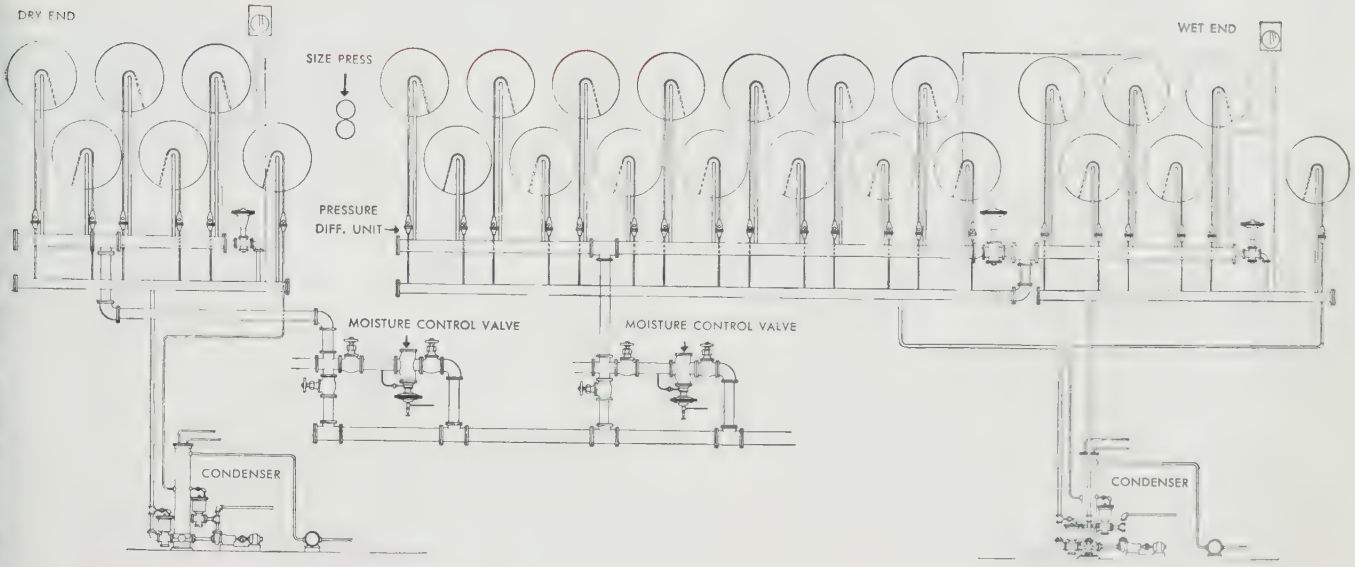
*Fig. 3*



Single 120-inch-coater (1960)

*Fig. 4*

# ALL Steamed Up OVER STEAM COSTS?

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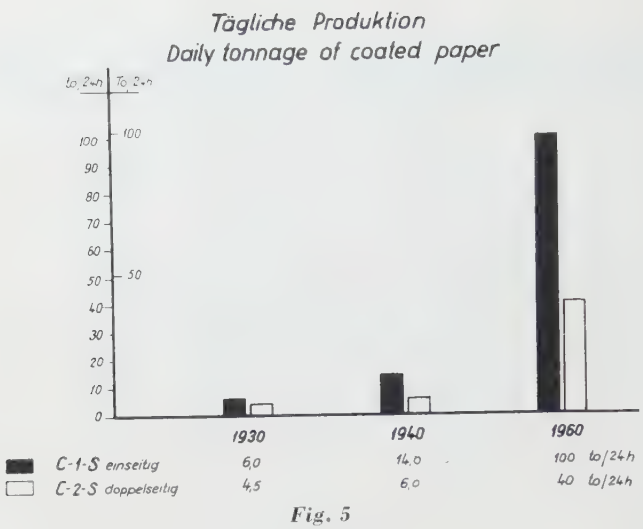


*Fig. 5*

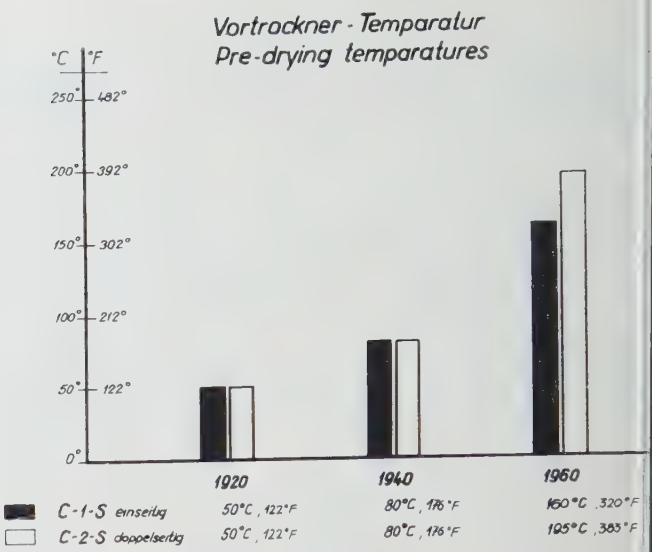


*Fig. 6*

production program consists of three furnish grades: wood free, 50% mechanical pulp, and label base paper.

In order to please our customers we are obliged to make 18 different basis weights, ranging from 53 to 200 lb. per ream coated, and we must produce up to 160 sheet sizes and 10 special shades. This huge variety of grades definitely makes life more difficult for us. It does not matter whether the coated paper is to be printed by letterpress, offset, or rotogravure. For several years our standard grade has been and is still used satisfactorily in all these printing processes. Figures 5, 5a, 6, and 6a sum up the changes in off-the-machine coating equipment within the past 30 years. Due to lack of information it is not possible to give comparative data relating to the development of off-the-machine coating machinery in the United States for the same period.

## FACTORS DETERMINING THE OUTPUT OF A 120 IN. WIDE DOUBLE COATER

That the output of such a wide coater is dominantly decided by the quality of the rawstock it has to coat was learned the hard way. By statistical analysis it was found that 80% of all the trouble, e.g. breaks, poor surface properties, trouble in the supercalenders, etc. is caused by the rawstock only. The uniformity in cross and machine direction of the paper to be coated in weight, thickness, moisture distribution, smoothness formation, wettability, and porosity are vital factor for a good coated quality. The degree of fineness o the groundwood, the blending of various chemical pulp as softwood, hardwood, and straw pulps and their degre of beating influence such things as machine speeds o coaters, surface properties, response to supering, dimen sional stability, curling tendencies in label papers, an penetration of adhesives in body stock just to mentio a few.

The rawstock has to suit the coating color and no vice versa. Therefore standardized coating color are applied to our rawstock and their composition never changed under mill conditions. A coated shee cannot be better than the rawstock from which is made. Therefore the superintendent of the pape mill is praised for the high tonnage and good qualit of the production of a coater and not the coatin superintendent.

To help him to achieve this the whole papermakin process has a very tight quality control system. A present four men per shift check, apart from the usua things, the phases of groundwood manufacture, pape machine performance at the wet end, e.g. sheet forma tion and retention on the wire. They also test shee

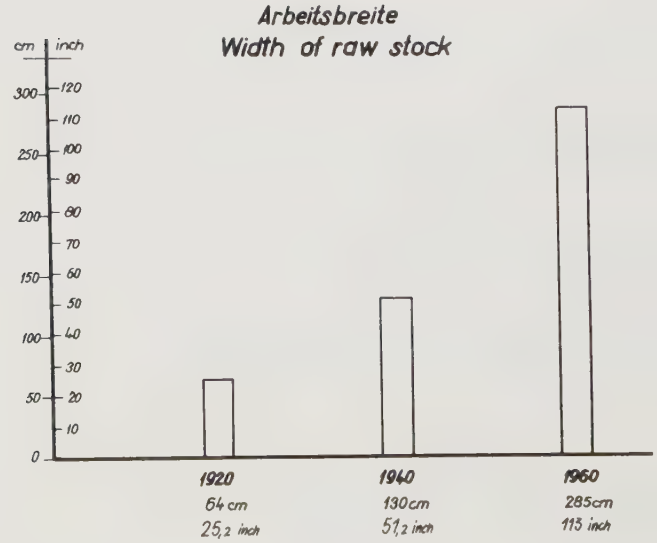


*Fig. 5a*

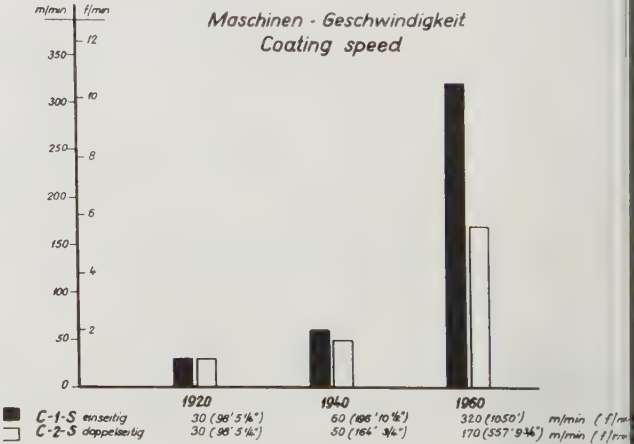


*Fig. 6a*

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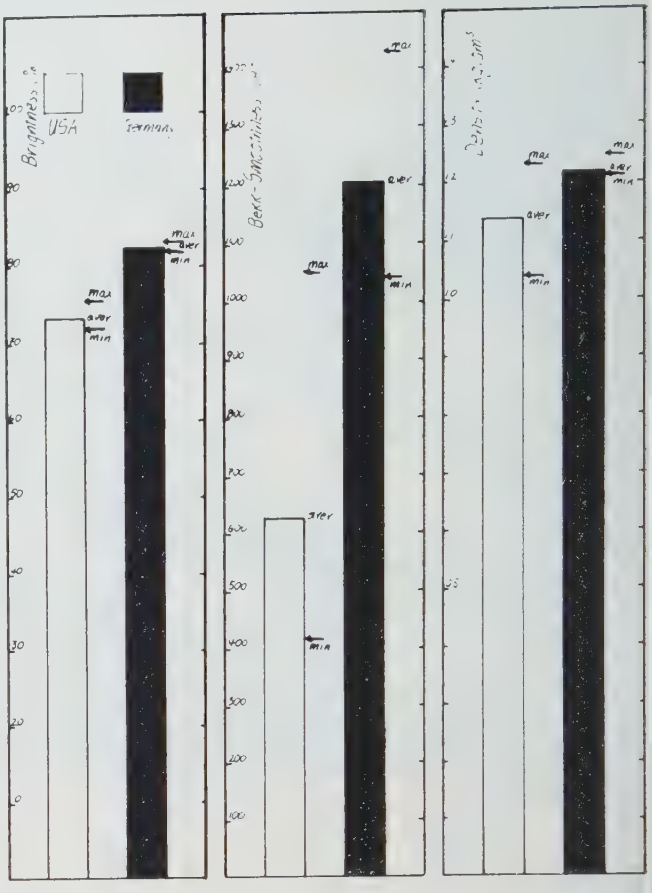
BASIC TO AMERICA'S PROGRESS

Woodfree coated Qualities

*Fig. 7*




Qualities with mechanical pulp coated

*Fig. 9*

profiles, each reel for imperfections, surface properties such as wire and felt marks, and make physical tests.

In 1960, 99.4% of all the paper made for the 120-in. coater performed well with an average of five breaks per day on this machine.

## COATED PAPER QUALITIES IN THE UNITED STATES AND IN GERMANY

Some data comparing similar grades of woodfree coated papers that are used in the United States and Germany for high quality printing work by off-set and letterpress are now given. They are all in the quality range of no. 1 and no. 2 grades.

Figure 7 compares these papers in brightness, smoothness, and density. These three paper tests were chosen as having the most constant characteristics to compare, because it is believed that other properties such as pick strength, ink absorbency, gloss, strength characteristics, or pH of coating have had to develop in different directions in each country in order to suit the printing techniques, the printing ink formulations, and public demand. The brightness was measured with an Elrepho-Zeiss instrument. Density is the quotient of basis weight and thickness of paper. It is regretted that it is necessary to use, for lack of anything better, the Bekk smoothness values. It is generally accepted that they are not sufficiently differentiating in a very

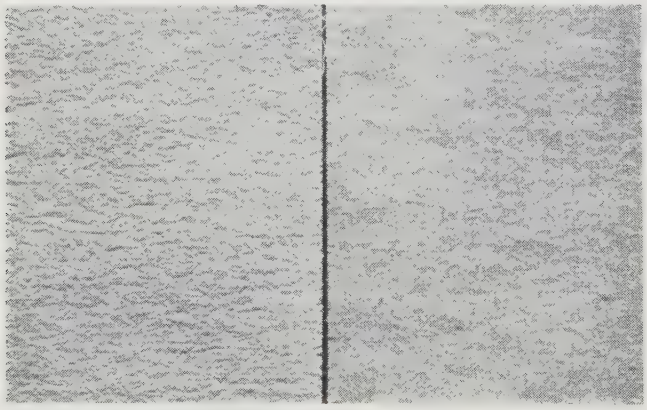


*Fig. 8*

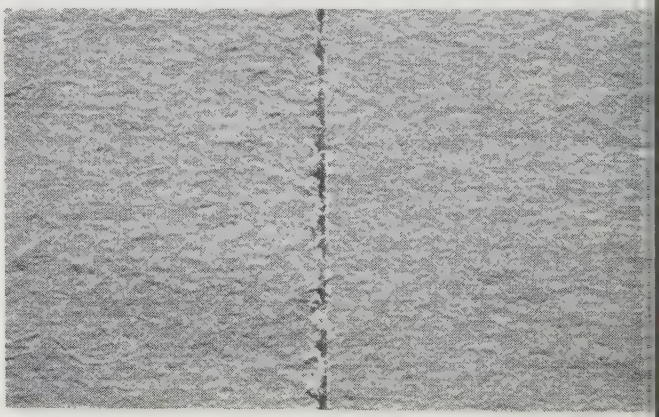


*Fig. 10*

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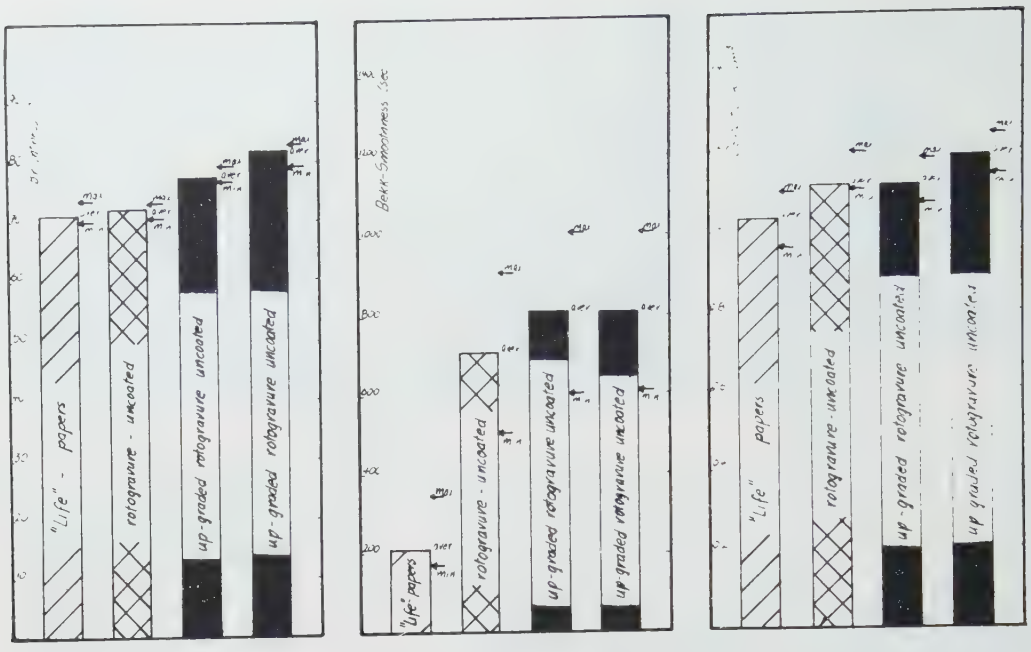


*Publication grades for letterpress and rotogravure*

***Fig. 11***

high smoothness range. It will be seen from the columns in Fig. 7 on the left that the average brightness of the American papers tested runs around 80.4. This average was obtained by testing 21 different papers. An average brightness of German papers was found to be 85.8, giving a difference of about 6 points in brightness. The average of the smoothness values arrived at for American papers was 550 sec. varying between 130 and 1400 sec.; the German average at present is 1100 sec.

The column on the right compares the density of these papers, and shows that in spite of the difference in smoothness there is only a slight difference in the average density values, i.e., American papers, 1.15 kg. per cu. dm. and German papers, 1.19 kg. per cu. dm.

Figure 8 shows photomicrographs of the surface of a woodfree American double coated paper on the left and an off-the-machine coated grade of Germany on the right. The difference in surface structure is apparent and the patterns of the roll coating processes are noticeable in the left picture.

Before trying to give an explanation for these findings, in Fig. 9 the test data for coated papers of the

***Fig. 12***

no. 1 and no. 2 quality grades containing me chanical groundwood i their base paper ar given. German qualitie contain up to 50% c bleached groundwoo Six American paper tested gave an averag brightness of 72.6 whe coated, the Germa qualities have brightnes values averaging 81.5 Here the difference i brightness is nearly 1( The smoothness averag again runs around 60 sec., quite close to th average of the woodfre coated papers of 550 se The average for Germa papers is 1195 sec. Th density values now a rived at are 1.13 kg. p cu. dm. for America papers, 1.21 kg. per c dm. for German papers. Apart from these data the is a more pronounced gloss in the coated quality Germany.

Figure 10 shows photomicrographs of the surfac of two papers containing mechanical pulp in the ba papers. On the left is the surface picture of a doub coated paper with 40% groundwood from the Unite States, on the right an off-the-machine coated gra from Germany with 50% groundwood conte Again a difference in surfaces will be noticed althou the surface of the American paper was trailing bla coated.

## HOW IS THIS DIFFERENCE IN BRIGHTNESS TO B EXPLAINED?

There seem to be three reasons for this differenc

*First:* Many German companies lost their chemic pulp facilities at the end of World War II and we forced to buy pulps for their paper mills on the wor market. Thereby they could benefit from the b advances that were being made in the pulp industri of North America and Scandinavia in pulp bleachi especially in sulfate pulp bleaching. The prese brightness demands could only be met by carel selection and blending of highly bleached wood pul by using bleached groundwood and the best fille available as English china clay, celites, and titaniu dioxide in the fiber furnishes.

*Second:* The coating colors, their adhesives, a pigments and additives had to be of the highest quali purity, and brightness. That is one reason why p ments such as blanc fixe, satin white, and the b English china clays are widely used in the Germ coating mills. An interesting consequence of this that, in general, coating of paper in Germany is mo expensive than the manufacture of uncoated pap because the cost of the coating color itself is higher th the cost of the fiber furnish it replaces. This prese a situation, which, according to what one gath from literature on coating, is the direct contrary

WATER COLOR PAINTING BY L. J. A. NEESE

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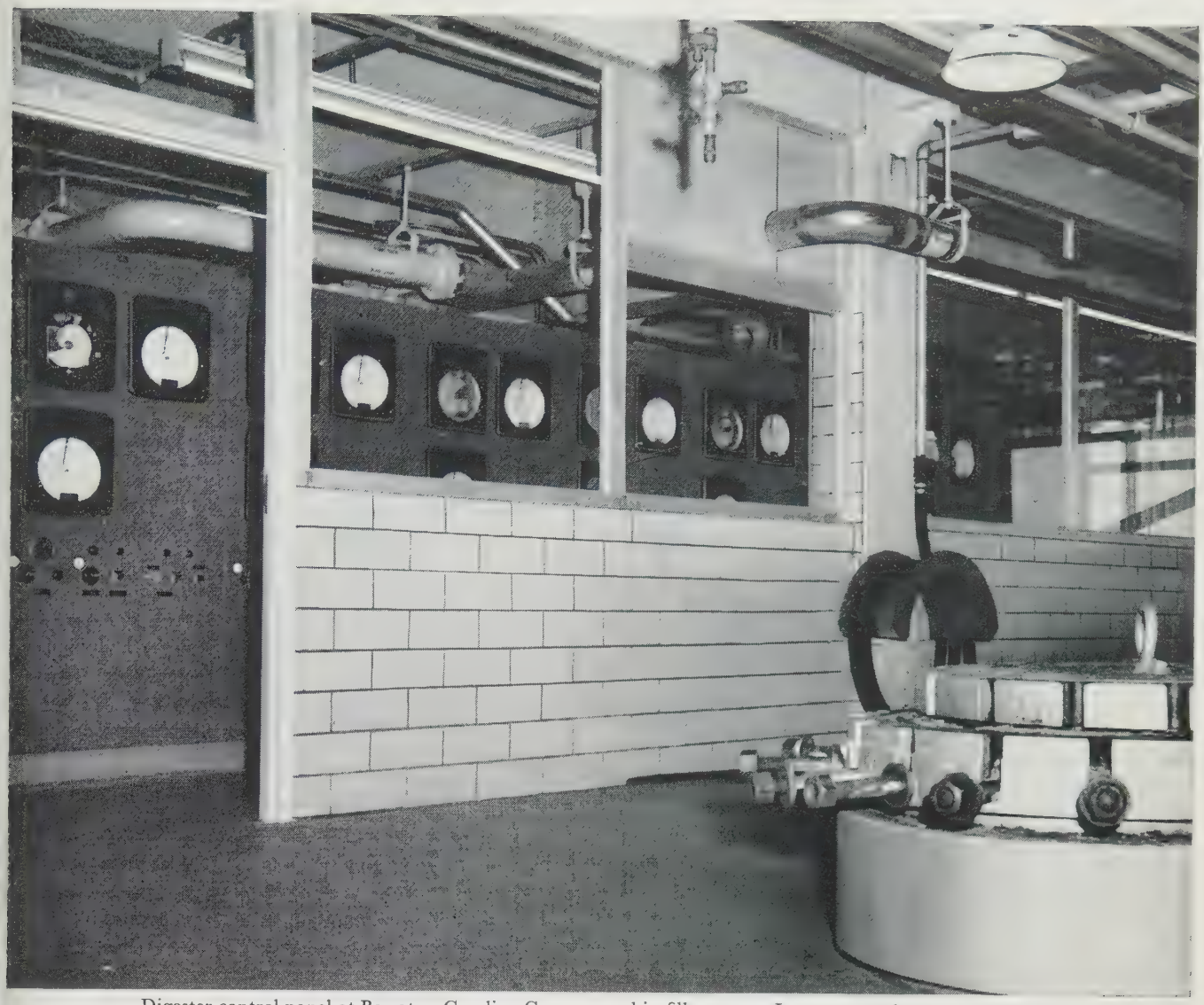

Digester control panel at Bowaters Carolina Corp., Catawba, S. C., containing 5 Foxboro Digester Control Systems, together with liquor-fill and chip-fill systems. Instruments shown are Foxboro cam pressure controllers, digester top-and-bottom temperature recorders, and relief flow controllers.

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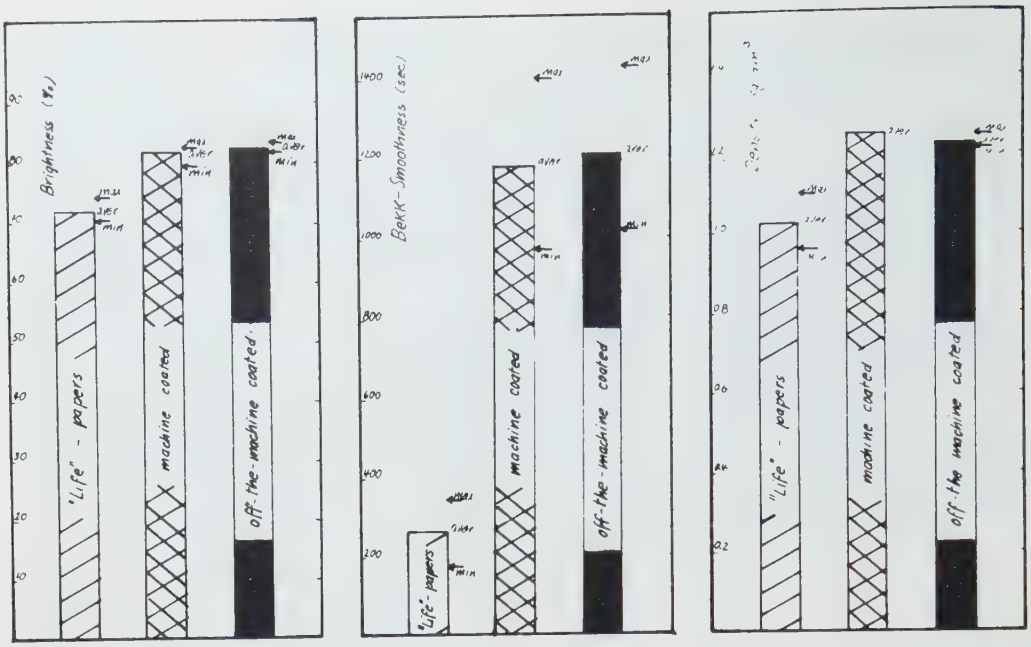



*The present level of machine coated papers among the other grades*

*Fig. 13*

that in the United States. Of course prices for pulps, pigments, and adhesives also have their part to play.

*Third:* All these endeavors would partly be in vain if the paper makers were not able to make a rawstock with as level a surface as possible. Fine machine wires, excellent sheet formation on the wire; good dandy roll operation: special press felts; and clean, smooth, drier felts: no calendar stack treatment in the paper machine, all these are vital prerequisites in order to do a good job and to produce a rawstock that will respond favorably to coating and supering techniques.

Again as regards costs, it is more expensive to make this rawstock than to produce uncoated grades.

## COMPARISON OF PUBLICATION GRADES IN BOTH COUNTRIES

Just as *Life* and *Look* are the popular magazines in the United States *Der Stern, Hör Zu,* and *Constanze* are published in Germany with up to 3.5 million copies per week.

While American publication grades are printed by letterpress the German papers are printed by rotogravure, and at present there is practically no market for letterpress publication in Germany. Similar to the

*Fig. 14*

upgrading already mentioned of the high quality coated papers these uncoated roto-papers have been greatly improved in the past years. Figure 11 gives data comparing these grades. On the left are the brightness, smoothness, and density of *Life* publication papers, data arrived at by averaging five test papers taken from various manufacturers in the States. The second section shows the present uncoated mass publication grades in use for German periodicals. The ash content of these papers runs around 20 to 25%. The ash content of *Life* coated papers was determined as 25%. Brightness is nearly identical, but because rotogravure printing asks for a leveler and smoother sheet, the smoothness of the roto-paper is as high as 700 sec. This type of paper is made on paper machines with widths up to 6 m. or 230 in. and with speeds of 500 m. per min. or 1650 f.p.m. The basis weight is 60 g. per sq. m. or 45 lb. per ream.

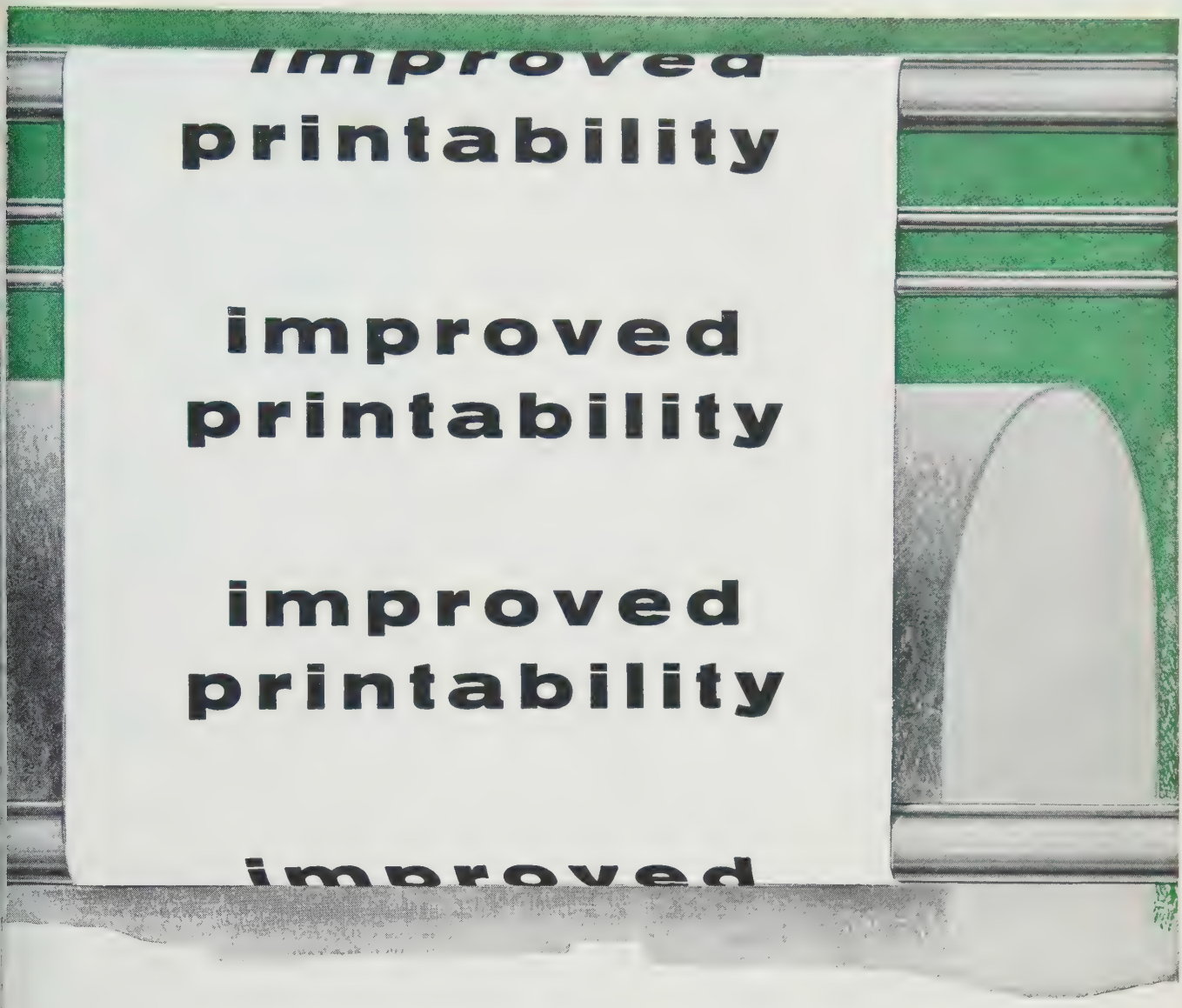
Figure 12 shows photomicrographs of a *Life* paper on the left and an uncoated roto-paper from Germany on the right. These pictures are self explanatory.

## THE FUTURE OF MACHINE COATING IN GERMANY

Bearing in mind the data on publication grades in both countries presented in Figs. 11 and 12, it will be possible to deduce that one of the main reasons why extensive on-the-machine coating in Germany has only recently become interesting is that the demand of the market for higher qualities was so far satisfactorily met at low prices by the producers of uncoated roto-papers.

Therefore, the three paper companies in Germany including the author's company, that have started on-the-machine coating had to develop a coated paper of similar quality to the off-the-machine coated grades in order to find a market. Figure 13 shows the quality level of these machine coated papers which are sold at present on the market. High brightness, good smoothness, and as level a surface as possible are their characteristics. These grades (Fig. 14) came just in time to fill a gap in the market that had arisen because the yearly growth in off-the-machine coating output was too small to satisfy the rapid market demands. But it is known that the big business lies with the publication grades, therefore all endeavors in the past have been and in the future will be focused on the task of opening a gate to this enormous market of the rotogravure publication industry in Germany.

At the 11th Coating Conference the same question was raised, so it seems that both countries are on the lookout for a successful solution of the problem how to make a good cheap coated sheet for the ro-

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gravure publication industry.

### THE FUTURE OF COATING IN GERMANY

In conclusion a few remarks on the future of on-the-machine and off-the-machine high quality coating in my country.

There is probably no doubt that coated publication grades will be and should be coated in the paper machine. But remembering the great strides in off-the-machine coating in the past years, there is a definite future too for off-the-machine coating. Mechanical engineers see no reason why off-the-machine coaters should not be able to run with speeds of let us say 1000 f.p.m. Although temperatures as high as 200°C. or 400°F. have been used for predrying no deterioration in quality has been noticed so far, and the final limit of temperature for this purpose still has to be found.

An excellent rawstock, high coat weights, and uniformity of quality are essentials for a high quality coated paper, therefore the challenge from the on-the-machine coating people in their efforts to break into the high quality market of coated papers can be faced calmly for the present. Automatic splicing, controlled moisture and conditioning, versatility with coating colors, and with trim widths are assets for the off-the-machine coating people that will pay off in the long run. Because the quality of the rawstock decisively determines the quality of the final product, it is the paper machine and the man who runs it, that decides whether the coated grade will gain and stay in the market.

With the closer contacts between the two countries in the fields of coating the author hopes that this comparison will be of advantage to salesmen, technicians, papermakers, and coating experts in understanding and appreciating the difference in quality of coated papers that are demanded by the present-day markets in the United States and in Germany.

RECEIVED April 11, 1961. Presented at the 12th Coating Conference of the Technical Association of the Pulp and Paper Industry, held in Buffalo, N. Y., May 8–10, 1961.

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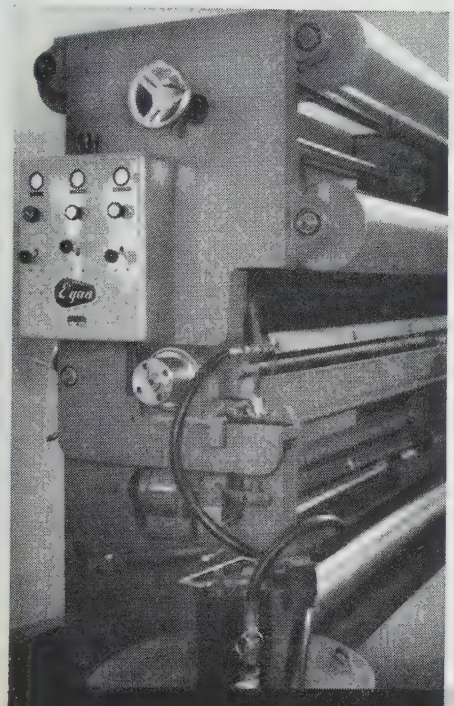

*This general view at left, depicts the Simplar's manually operated impression roll control wheel with leveler knob, as well as the cylinder bearing "skirt support" with its pneumatically controlled toggle for "on-off" impression.*

*The complete three unit Simplar installation pictured below handles a 60″ wide web. It includes an Egan Phantom-Axis Turret Unwind, infeed system, and Egan High Velocity Dryers. The dryers have a temperature range up to 450°F. with 100% make-up air.*

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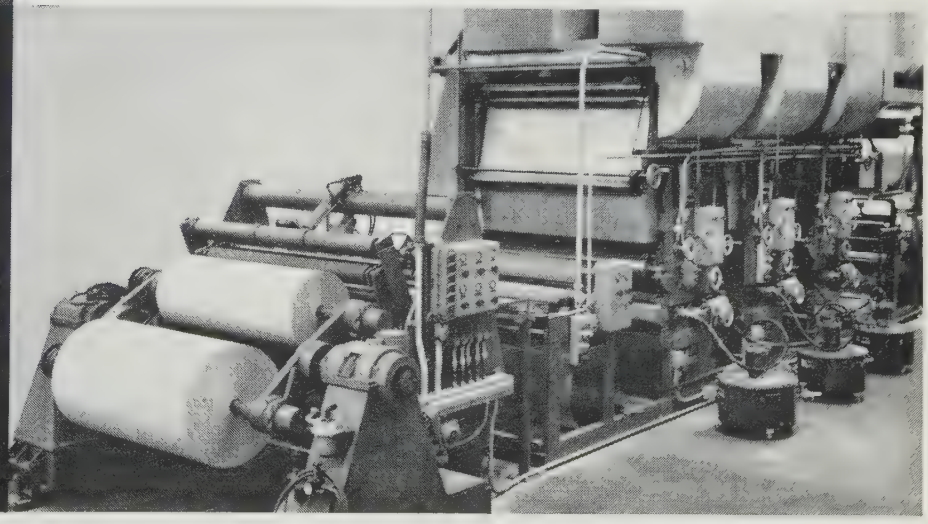

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*The Magnefite process* has been successfully applied to a wide variety of woods—jack pine, tamarack, white pine, birch, elm, poplar, maple, and other difficult-to-process woods—permitting a lower cost/ton of pulp produced.

*Processing advantages are numerous.* Cooking cycles are shorter since there is no limit to the speed at which the temperature of the digester can be raised. The absence of large quantities of $SO_2$ gas in the digester permits cooking at higher temperatures. A rapid increase in temperature is also possible as a result of impregnating pulpwood chips with hot magnesium bisulfite liquor of equal combined and free $SO_2$. With such rapid penetration of chips, cooking time is reduced as much as 40%! This results in a substantial increase in digester capacity. The absence of true free $SO_2$ simplifies liquor make-up, eliminates the need for pressure storage vessels, and practically does away with dry relief.

In some cases, the cost and processing advantages of Magnefite pulping justify its use even without chemical recovery. Where chemical recovery is justified, 90 percent of the pulping chemicals may be reclaimed and reconstituted in a recovery system.

Dow is a basic supplier of the principal ingredient, magnesium hydroxide . . . in slurry form for maximum handling ease . . . available for immediate delivery. Chances are Magnefite pulping can be an advantage in your processing operation. Write THE DOW CHEMICAL COMPANY, Midland, Michigan, Chemicals Sales Department 802FC12, for further details.

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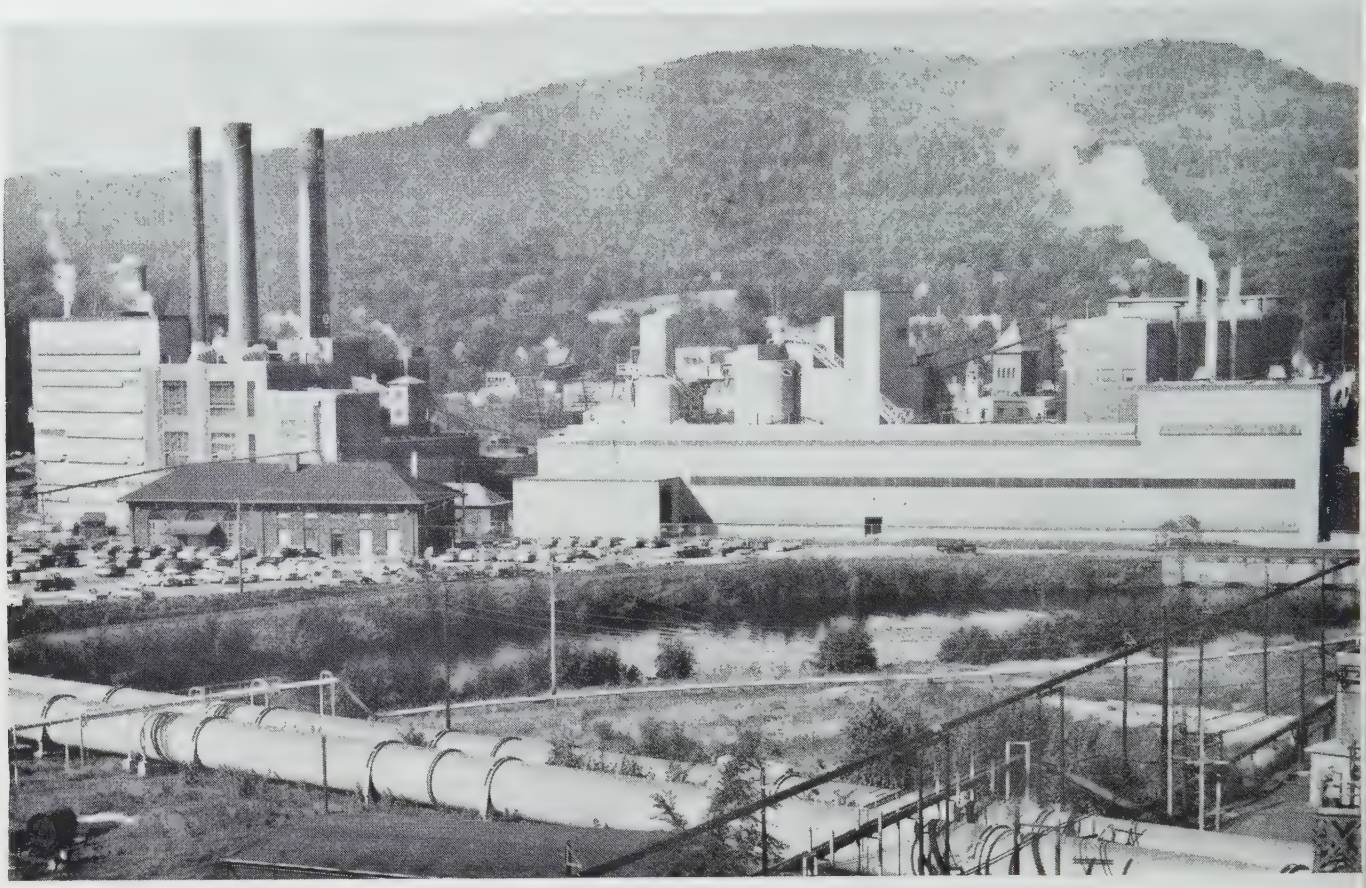
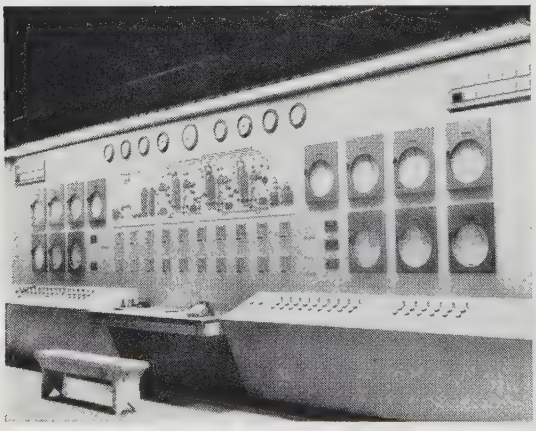
**THE DOW CHEMICAL COMPANY** DOW Midland, Michigan



# RUST helps expansion program at Oxford



• *Bleach plant control panel designed and erected by Rust.*

In addition to the expansion program work by Oxford's own engineering staff and construction department, several new pulp mill buildings at Oxford Paper Company, Rumford, Maine, were designed and constructed by The Rust Engineering Company. These buildings include the slaking and causticizing plant, chlorine dioxide manufacturing plant, hardwood kraft bleach plant, chip silos and lime recovery plant.

Working in conjunction with Oxford's own staff, Rust designed the soft wood bleach plant and recovery boiler, which were built by the Oxford construction department.

Effective teamwork resulted in the expansion program being completed on schedule.

**THE RUST ENGINEERING COMPANY**

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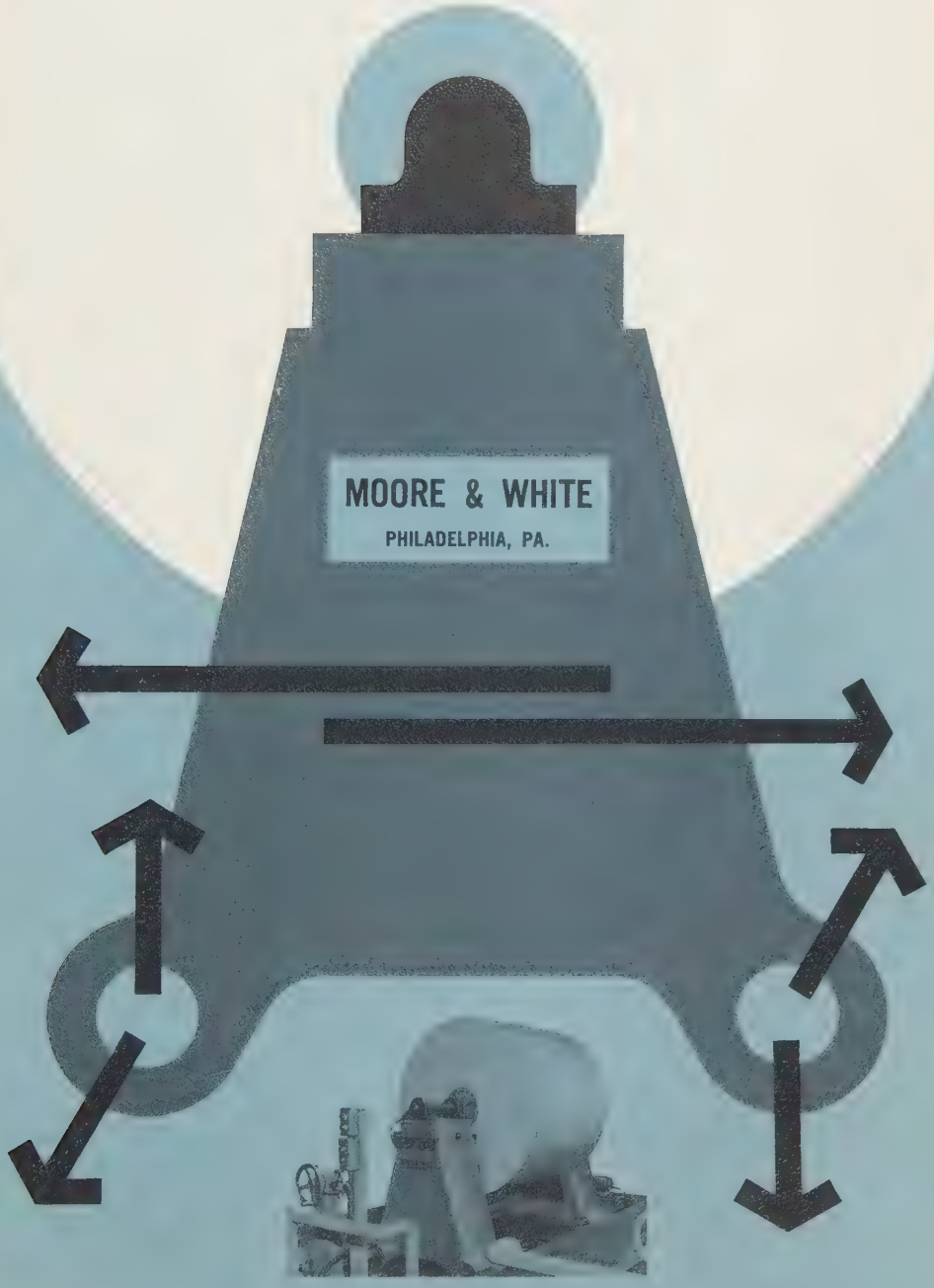
P-10

# THE MOORE & WHITE SHAFTLESS BACKSTAND

increases available working time and provides greater flexibility for a wide variety of web fed equipment such as cutters, rewinders, laminators, etc.




- Easy roll handling, one-man operation without special labor, overhead cranes, lift trucks, etc.
- Handles a variety of roll widths and sizes.
- Complete power adjustment.
- Automatic tension control.
- Effective pneumatic braking.

THE MOORE & WHITE COMPANY

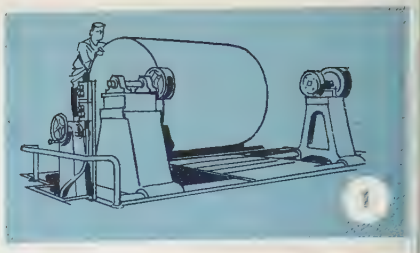
## Easy roll handling by one man with

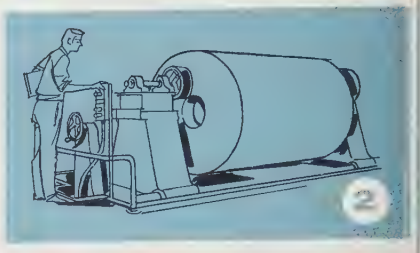
# The MOORE & WHITE
# SHAFTLESS BACKSTAND

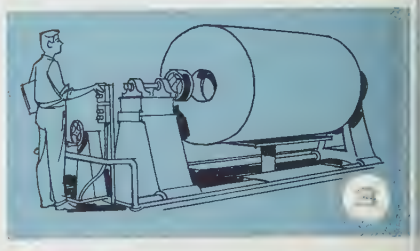
Roll is pushed on floor tracks or rolled (1) between the end stands where a hydraulic lift (2) raises the roll into position (3) The end stands are then run (motor driven) into position until pneumatic chucks engage and lock the roll on the spindles (4) A large size pneumatic brake (5) controls unwind tension and stops the revolving roll automatically and instantly in the event of a paper break. Easy adjustment in all directions (6) assures that any roll will run true. Extra heavy duty construction assures trouble free operation.

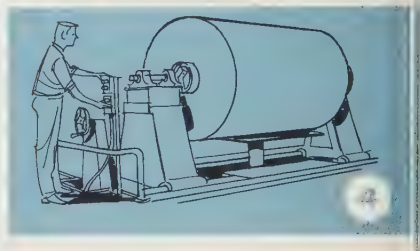
A series of Moore & White shaftless backstands provide multiple feed or successive feed for a wide variety of converting equipment. For example, three stands can feed lightweight grades simultaneously. When successive feed is needed, maximum utilization can be obtained by running the back unit first, followed by the second. Then, as the roll nearest the machine is being run, the other two are reloaded. The process is repeated in the same order; while the roll on the middle stand is run the back one is reloaded. Run this way, a three stand arrangement permits a single operator to run 6 rolls without downtime for reloading. Users report working time and flexibility equal to or greater than that realized with shuttle type duplicate backstands.

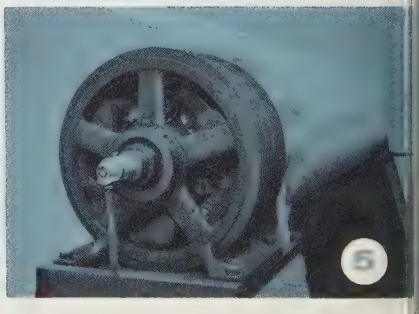
You can save time and money with the greater machine utilization possible with Moore & White Shaftless Backstands—designed and produced to fit your special needs by



## THE MOORE & WHITE COMPANY

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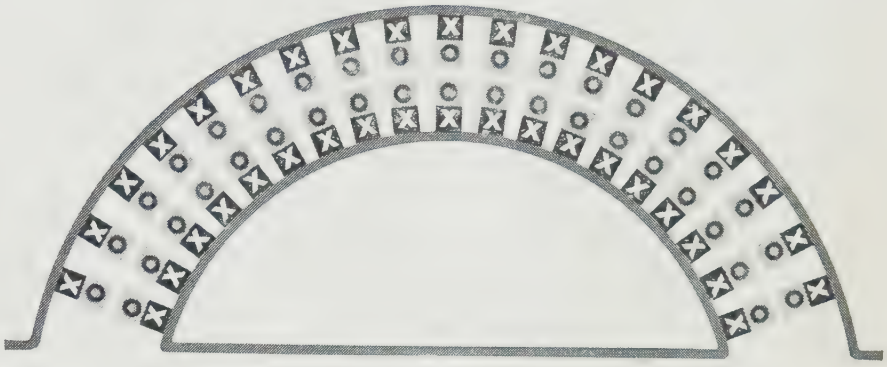
78 YEARS

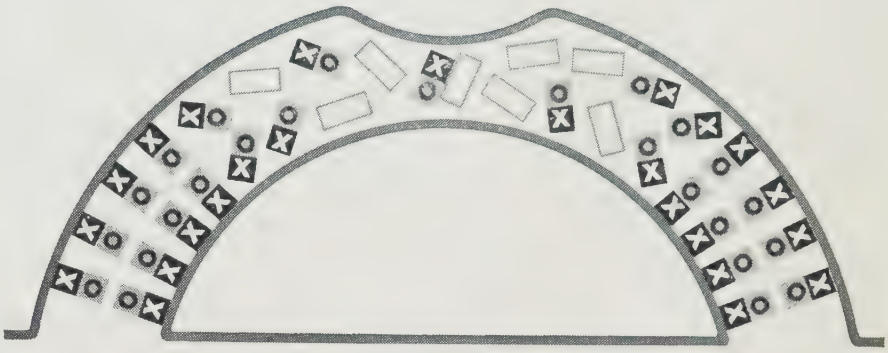
# The make-up and break-up of foam

Foam, of course, is made up of bubbles. But, all bubbles do not form foam. For instance, pure water will not foam. If you take a glass or a beaker of pure water and bubble air through it, the bubbles will be large and will rapidly rise to the surface and burst. Several smaller bubbles may even join together and rise to the surface. But even these burst at the surface. There is no tendency to form foam.

Now, add just one drop of detergent—a surface active material—to this same water, blow air through it and foam develops. The bubbles are relatively small and there is no tendency to coalesce. This foam-causing difference is due to the surface activity of the detergent molecules.



**Bubble Cross-section**



**Bubble with Defoamer Added**

### The Make-Up of Foam

Each surface-active molecule has a "water-loving" end and a "water-hating" end. These molecules arrange themselves in an orderly fashion in the liquid film of the bubble with their "water-hating" ends toward the film surfaces and their "water-loving" ends buried in the liquid interior of the film. It is this arrangement of molecules that gives strength and elasticity to the bubbles and keeps them from bursting.

### The Break-Up of Foam

Anti-foam agents act either by completely displacing the surface film or by modifying its film-forming properties so that it no longer forms a stable foam. Therefore, a good defoamer must be capable of entering the liquid interior of the bubble film and modify or break-up the molecule arrangement that sustains the bubble film. Thus, since the defoamer only has to work on only a small quantity of surface active material, only a relatively small amount of defoamer is needed.

### What Causes Paper Mill Foam?

Some impurity, intentional or otherwise, must be present before foam can be produced. The most persistent foams are produced when the impurity is a surface active material such as carbohydrates, lignin, dyes, black liquor soaps, slimicides, sizes, etc. Since a foam condition can be caused by any one—or several—of these materials, no single defoamer will fill all needs economically. Houghton's "Personalized Paper Mill Service" was instituted to provide this "diagnosis and curative" service with proven De-Airex defoamers. If you have a foam problem in your mill, call your Houghton man or write and request this technical help. It's yours for the asking and may save you untold headaches and expense. E. F. Houghton & Co., 303 W. Lehigh Ave., Philadelphia 33, Pa.

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# The Electronic Computer "It Counts"

F. A. REED

**The history of the computer is recounted and the basic operations of a modern computer are described. A set of orders, called a program, is transferred to the unit, usually by means of a typewriter, on tape or punched cards. The computer then works by application of a binary code. An outline is given of the range of functions that can be performed.**

## ANCIENT HISTORY OF COMPUTERS

THE FIRST computer, as history records it, consisted of pebbles laid in furrows drawn in the sands of what is now the Middle East. Pebbles corresponding to the first number to be added were placed in the furrows with the right-hand furrow corresponding to the units digit of number, the next furrow to the left corresponding to the 10's digit, etc. (Note that the development of a number system wherein the value of the digit is affected by its location in the number had already been developed. This development made mathematics as we know it today possible.) As the other numbers to be added were likewise represented by the pebbles some furrows became filled to their capacity of 10. The pebbles were then removed from these furrows and the value of 10 was represented by one pebble in the next furrow to the left. When all numbers had been represented the sum could be read by counting the pebbles in each furrow.

The device was later transferred to clay tablets for portability and, still later, with some modification, was made into a frame with beads strung on rods which is the abacus used extensively in many parts of the world today.

Man progressed from there, as his mechanical and mathematical ability increased, to many mechanical counting, adding, and arithmetic devices, even including some to do the mathematics of differentiation and integration. These devices were in the early days operated by hand power and were later converted, for ease of operation, to electrical power.

## MODERN COMPUTERS

It was not, however, until about ten years ago that sufficient knowledge in electronics and mathematics was accumulated to make possible the construction of an electronic computer. The first electronic computers employed vacuum tubes for circuit switching and due to the tube size and heat generated were quite large and required special cooling. An additional difficulty encountered was the relatively short life of the tubes. Repairs were frequent and expensive, and reliability was poor.

This picture changed with the development of the transistor. This device replaced the vacuum tube and eliminated most of the complaints of the tube computers. Size was reduced to that of an office desk, most heat was eliminated, and reliability became excellent.

As manufactured today an electronic computer is a machine which, through the use of transistors, resistors, condensers and magnetic forces, achieves the ability to add, subtract, multiply, divide, receive information, store information, and disseminate information. These functions can be done at an amazing speed and with extreme accuracy, because of the elimination of all moving parts within the computing components. All current switching and logic decisions are made electronically and at an electronic speed.

High speed internal operation in itself, however, is not sufficient to provide overall high speed computing. Operations can only be executed as rapidly as they are presented to the machine, and if this were done by an operator (as with an electric desk calculation) the maximum speed would be limited to the speed of the operator. This limitation is overcome by using the storage ability of the machine. The set of orders necessary to solve a problem is "read" into the machine and stored as magnetic "bits." These orders, called a program, are transferred one at a time by the computer to its operation registers and the function called for is executed. The program is not destroyed by this operation and so may be repeated as frequently as necessary. The machine is now relieved of the slow actions of its operator and can perform its task step by step as rapidly as it can present the predetermined program to itself.

It is the program stored in the calculator's memory that determines what the machine will calculate. The machine itself is general purpose and can compute anything that is mathematical and many things that are not.

For this reason, the machine is no wiser than the program that controls it. To say, however, that the machine is no wiser than the programer who wrote the program may not be correct as some programs can learn by experience, but more about that later. The program controls the machine so it is inaccurate to say, for instance, that an electronic computer failed to predict the outcome of an election. It was the program that failed, not the computer; that is to say the series of steps performed or the data considered by these steps caused the error, not the computer. It is this program that provides the great versatility of these machines—design a bridge—play chess—compute a payroll—calculate the orbit of a satellite.

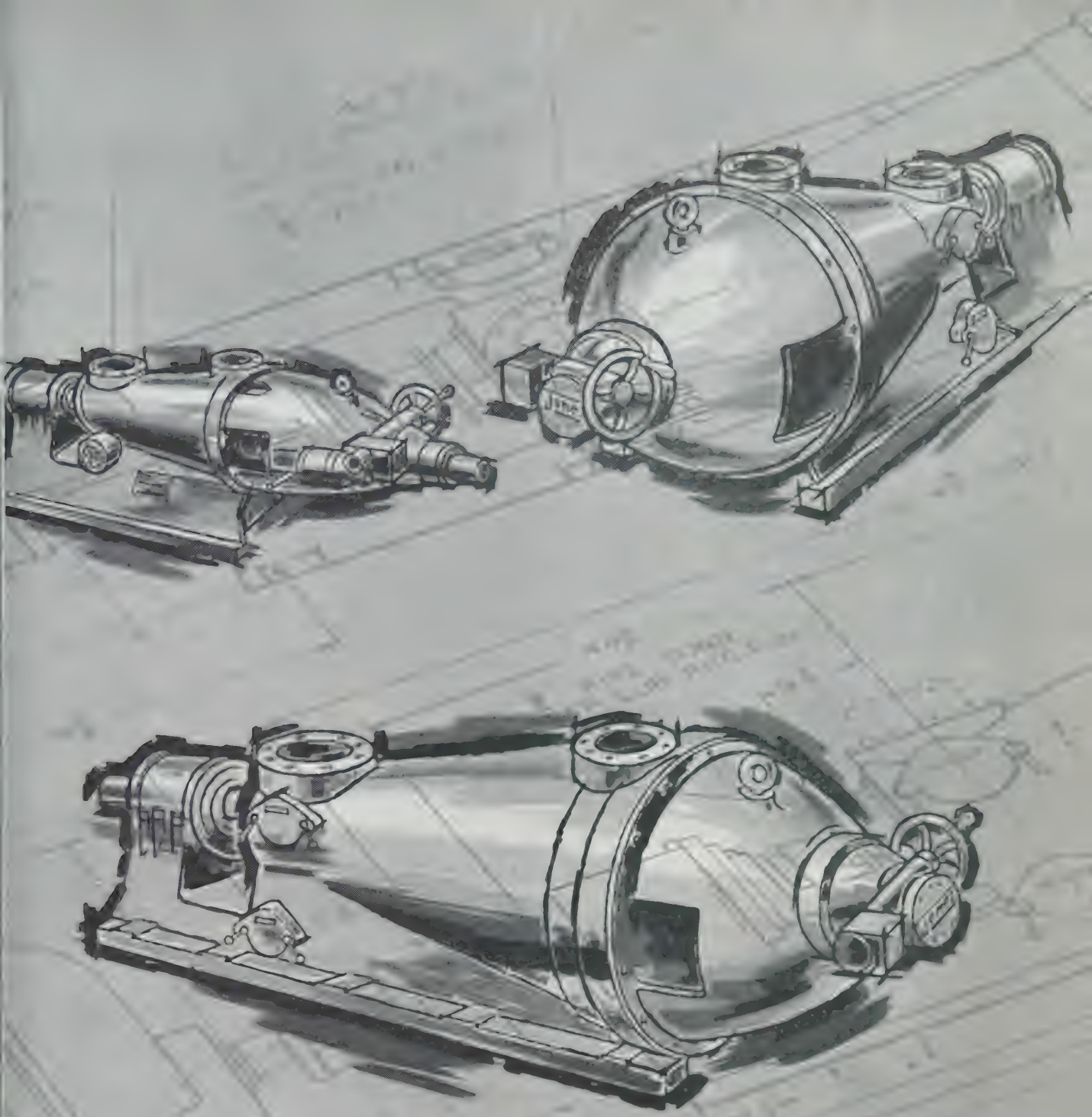
## MACHINE COMPONENTS

What components does a computer need to solve problems? The operations needed are as mentioned briefly above: input, output, storage, arithmetic, and logic. The hardware needed may come all in one machine or additional peripheral equipment may be needed for a particular type of problem. Generally, input and output may require extra equipment and storage, arithmetic and logic are provided in the basic machine.

Input and output may be accomplished in several ways and at greatly varying speeds. The basic input-output on most computers is the electric typewriter. The typewriter is under operator control for input and under computer control for output. The input can be for most computers, numeric—consisting of numbers, or alphameric—consisting of numbers, letters, or special characters. (Strangely enough modern computers can handle letters just as easily as numbers but the "mathematics" of letters is not as well developed as the mathematics of numbers.) Speed of input or output on the typewriter is always very slow compared with the speed of the computer. Input speed, controlled by the operator, is zero to ten characters per second. Output is at the maximum speed of the typewriter usually between ten and twelve characters per second. This does not compare favorably with internal calculation, where the addition of two digits may take only one-thousandth of the time required to print the result.

To speed input to the machine, punched paper tape, punched cards, magnetic tape or magnetic disks are available. The information stored on these devices must all be prepared

F. A. REED Computer Section, The Rust Engineering Co., Pittsburgh, Pa.



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in advance either by hand or by computer, but may then be entered into the machine at high speed. Paper tape will read (enter information into the machine) with speeds of 50 to 500 characters per second; punched cards at speeds up to 330 characters per second; and magnetic tape or disks storage up to 90,000 characters per second.

For output, stick or line printers are available with speeds up to 1000 complete lines of print per minute (about 1300 characters per second).

## HOW THE COMPUTER WORKS

Storage in electronic computers is based on the retention of "bits" usually as spots of magnetism or in some machines by electrostatic charges. These bits are considered in groups by the computer and represent the number or letter in binary code. The groups of bits are stored in individual locations each of which is numbered with an address number. This makes it possible to store or retrieve any piece of information at will. These locations can be used to store data, results, coded letters or the program instructions. The use to which the program puts the location determines which operation it performs. It is through the ability of the computer to store or retain its own information that high speed calculation is possible.

Binary code, which is used in some form by all electronic computers, is the representation of numbers or fractions by sums of powers of two. All whole numbers can be represented and the reciprocals can represent fractions covering the full range of fractions. Thus $2^0 = 1$, $2^1 = 2$, $2^2 = 4$, $2^3 = 8$, etc., would represent numbers that are a direct power of 2 and the figures 3, 5, 7, etc., would be represented as sums of the powers: that is, $2^0 + 2^1 = 1 + 2 = 3$, $2^0 + 2^2 = 1 + 4 = 5$, etc. The binary code is written with only two symbols (ten are required for the decimal system), usually a one and a zero. The one indicates a power of two and a zero represents zero. Any bit has twice the value of the bit position to its right so that a number written as 1101 with the rightmost digit in the units position would represent 13. This is the sum of the value of the bits or $8 + 4 + 0 + 1$. Letters are represented by a code and are decoded by the computer circuitry. Thus 41, 42, 43, etc., may mean respectively—$A$, $B$, $C$, and 23 might represent a comma.

Arithmetic is accomplished by one of two general methods, both of which stem from the computer's ability to add. The first method which is used on all machines that use the binary system for internal storage is a result of the binary system itself. That is, for example, 1 bit + 1 bit (01 + 01) equals no bit + one bit to carry resulting in an answer of one bit — no bit (10). This configuration in binary indicates a 2. This operation can be carried on by electronic circuits using a relatively simple flip-flop switch.

The second method used on machines that store their information internally in the decimal form is accomplished by a table lookup, just as a person would do if he could not remember that 2 plus 2 is 4.

The other arithmetic operations performed by the machine are the result of its ability to add. Subtraction adds the complement of the number. Multiplication adds repeatedly to form the multiplicand (or uses a table lookup). Division subtracts repeatedly to form the quotient.

In all arithmetic operations, the sign of the number must be determined for the answers to be meaningful. This operation in an electronic computer is accomplished by a bit or lack of a bit accompanying the number to signify the sign. Special circuitry handles this sign bit according to the algebraic sign rules.

Logic is the ability of the machine to make a choice as to the operation to be performed. For example, let us say that it is desired to obtain a value for $Y$ from one of two equations, $Y = A + B$ or $Y = C/D$, depending on whether the value of $X$ is greater or less than 100. This operation would be performed in the computer by computing the value of $X$ and then comparing it with 100. If $X$ is greater than a hundred the computer would compute $Y = C/D$, if less, $Y = A + B$ would be computed and the appropriate value of $Y$ would be stored for future use or for print out. The compare operation is done by subtracting the value of $X$ from 100 and branching on whether the result is negative orpositive.

This logic also permits loops to be constructed within the program wherein the same operation, or set of orders, is performed repeatedly. The value of numbers being operated on in the loop may be changed before entering the loop or the number of times the loop is executed may be altered by the calculations. A simple example of this would be to multiply $3 \times 1$, $3 \times 2$, $3 \times 3$, etc., that is, make up a multiplication table of threes. The basic operation would be to multiply two numbers together. Before entering the loop the first value would be set to three and the second set to one. After the first loop the second value would be increased by one and the loop re-entered. The execution of each loop would then provide an answer—3, 6, 9, 12, etc. These values could be stored or printed out by additional orders within the loop.

The loop as outlined above would continue to be executed until stopped by the operator. The operation could, however, be terminated within the program by adding instructions to stop when the second value had reached a predetermined value or this value could be determined by other considerations within the program.

It is through the use of this logic that the program obtains its ability to execute extremely complicated and versatile problems.

In this type of programing all loops or branches of the program are set up by the programer and are either used or not used by the program, according to the requirements of the problem.

## WILD BLUE YONDER

There are, as mentioned earlier, certain programs, mainly experimental, where the actual program itself is changed by the program to accomplish additional tasks not contemplated in the original program. This type of program usually proceeds on a random or trial and error basis and weeds out the bad from the good by some criterion determined by the problem.

An example of this might be playing a game on the computer where the rules define a winner and a loser. Actions in the game which contribute to winning are preserved by the program and actions which lose are discarded. In this fashion the program can become more and more experienced by playing more games.

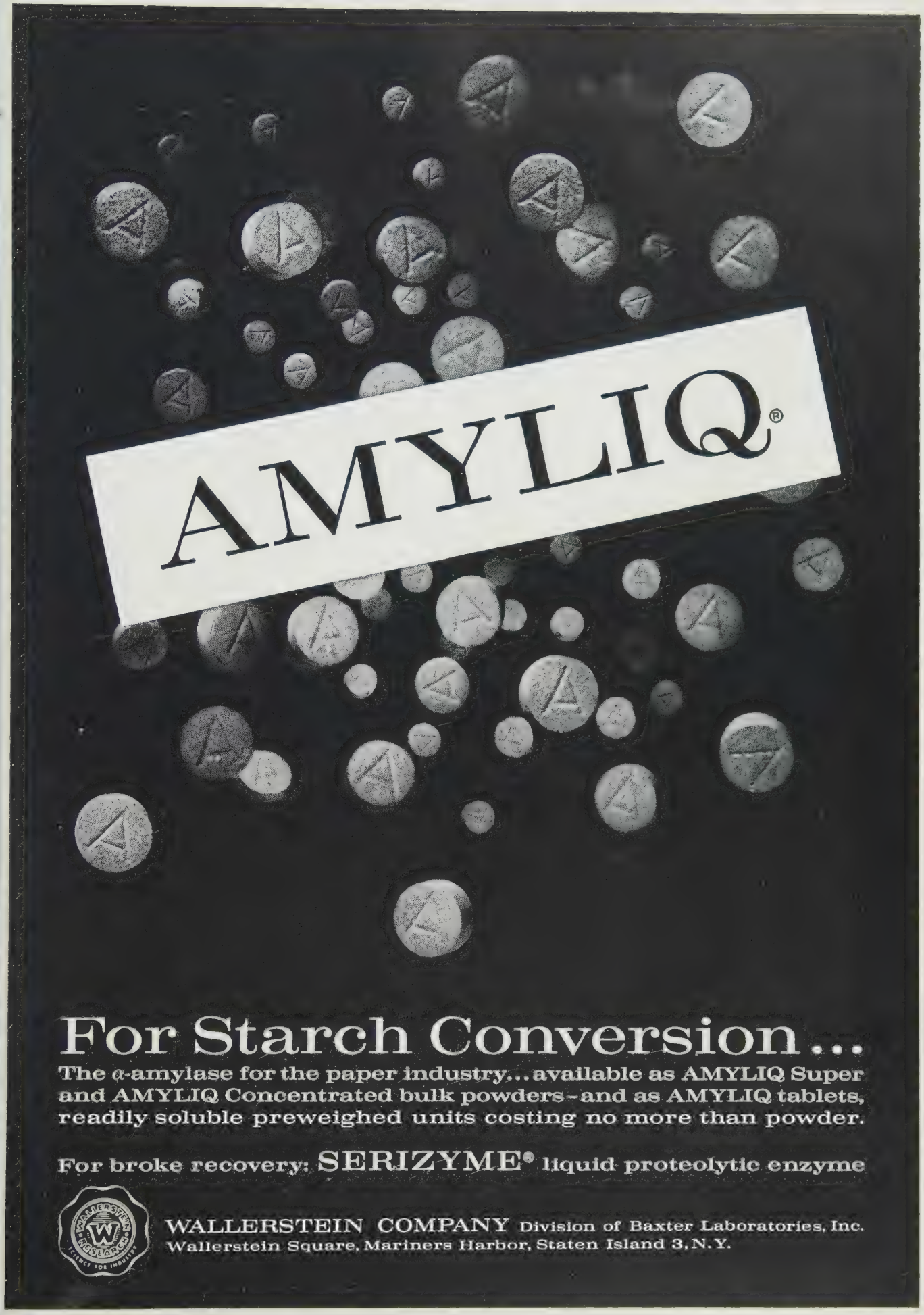
It can be stated with reasonable certainty that any mathematical calculation that can be done by hand can be performed on an electronic calculating machine. The only real limiting factor is economy.

The fields of accomplishment of the computer consist of accounting, payroll, cost control, inventory control, transportation and distribution problems, production scheduling, machine design, building design, machine control, quality control, and machine simulation; also, to a limited but growing extent, the ability to make management decisions and help train management to make better decisions.

You say, if the machine can do all of these things it must think much the same as humans do. The arguments on this point have been raging for some years now.

What is thinking? Do you expect a baby or an uncivilized person to match the thinking power of a university professor? If not, why not? "Training," you say! The professor has had long years of training and has accumulated a vast store of knowledge and an even greater amount of knowledge stored in books readily available to him.

I change the terminology and say the professor has been programed to do many things, has many facts in his high

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speed memory and many more facts in his slower but larger magnetic disk or tape memory. But you say the professor has developed a proof of a theorem never proved before by man, that he has created this from his thinking power. I say, an electronic computer program has proven a theorem in a better and shorter fashion than man has done before.

You are probably saying to yourself, "But the professor was thinking and the machine wasn't, it couldn't, machines can't think."

Let's explore thinking just a little more. Take tic-tac-toe, the game played with X's and O's. If you have never played the game and know nothing at all about it, how would you learn to play it expertly? There are two ways. You could get an expert to teach you the rules of the game and the rules for playing expertly. He could also coach you, point out your mistakes, and allow you to acquire a great deal of experience in playing with an expert; or, you could read the rules that determine the game and the winner or loser and then just start playing, sorting out the good moves from the bad by whether they lead to a win or a loss. This way, in time, you could become a self-taught expert.

The same approach can be used in writing a program for an electronic computer. All the game rules and the rules for playing expertly can be built in to the program and you have an instant expert; or, the game rules only can be built in and the plays selected at random with the good plays being retained and the bad plays discarded. In time, you have a program that can play expertly.

There is a striking resemblance between the processes employed by a human and the processes used in the electronic computer.

However, you are probably still saying to yourself, "a machine can't think because,—well, a machine just can't think."

Perhaps the solution to this argument will be to redefine "thinking" to include the phrase "by humans."

## WHAT COMPUTERS CAN DO FOR YOU

A paper mill has business functions as well as production functions and so becomes involved in accounting, payroll, and cost control activities. These can all be handled on an electronic computer as is now being done in increasing numbers by business concerns of all types.

The problems involved in accounting and payroll calculations are characterized by the vast amount of information that must be handled both as input and output and by the simplicity of the calculations required. This necessitates the use of a computer with high speed input/output. The program, however, is usually quite simple as compared with say, engineering programs. The exception to this case may be the testing required to handle special cases. These can, of course, be programed and handled on the computer, but may not be worth the added programing time or the additional running time for the program.

Many accounting systems, and this applies to other procedural systems as well, that have been developed over a long period of time are studded with special cases, sometimes to the extent that the normal procedures have actually become less used than the special cases. It is also frequently found that parts of reports prepared for management are duplicated in various forms by one or more lower echelons. Considerable money can usually be saved by preparing these only once in a standard form usable by all. This points up the fact that it is frequently wise to overhaul the whole system when converting to machine operation.

The main advantage in using an electronic computer in cost control is the speed with which records can be compiled and reviewed. Programs can be written so that danger areas or borderline cases will be flagged so that little time need be spent on areas that show no difficulties. Reports that formerly were compiled monthly or quarterly, if desired, can be on management's desk each morning. This makes it easy to spot trends and make corrections in operating procedures before damage can result.

The paper industry can expect to draw heavily on the experiences of other industries in the use of computers to solve the problems of inventory control, transportation and distribution, and production scheduling, as these problems are almost identical, regardless of the product manufactured.

Electronic computer inventory control makes it feasible to handle large inventories of material in stock that can be produced during slack time and thus smooth out the peaks in the production schedule. A complete inventory can be kept on the computer and be continuously revised during the day to show material in stock, material entering storage, and material being removed from stock. However, this is not all; the machine can also process orders and pin-point which rolls or sheets an order should be cut from in order to reduce waste. In a matter of minutes it can try every roll and sheet in storage to find the optimum cutting procedures. Trim sold to the customer is extra production.

Transportation and distribution problems can also be solved. One large canned food company has recently increased its profits by a half million dollars a year just by the use of a computer and linear programing.

If more than one mill or warehouse is involved in the distribution of the final product to the customer, it is probable that linear programing can produce a better and more economical routing than can be done by usual methods. This is particularly true if more than one method of transportation is available and if shipping rates change.

The movement of raw materials into a mill or to storage from several sources and by different modes of transportation can also be economically planned by a computer.

In these cases, as with many other uses of the computer, one of the big advantages is the speed with which a new plan or schedule can be produced when changes make it necessary.

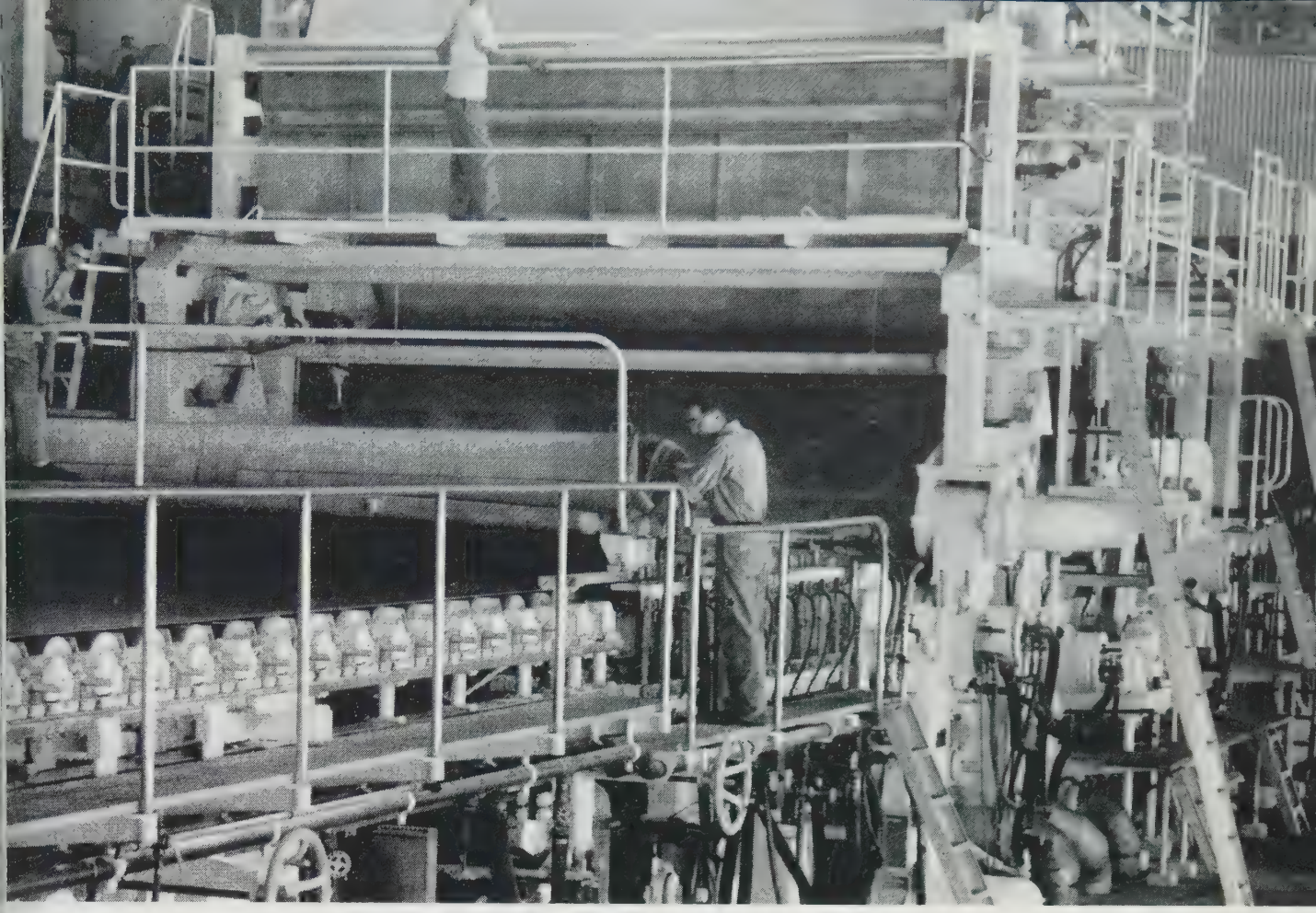
Production scheduling can be a problem: Which machines can most efficiently and economically produce the orders on hand, under contract or anticipated? What effect does the reduction in production width, due to felt wear, have on the present schedule? If a large new order comes in, should the present schedule be maintained or should changes be made? A computer, by the use of linear programing, can solve these problems quickly and accurately.

What is linear programing? It is the solution of several linear (straight line variation) simultaneous equations for a maximum or minimum value when the number of unknowns exceeds the number of equations. To aid in the solution, certain restraints must be placed on the unknowns to reduce the number of possible solutions. An example of a restraint might be that the quantity of a material purchased cannot be negative; that is, you cannot buy less than a zero quantity.

An example of the use of linear programing might be to determine the number of items to carry in stock so that the profit would be a maximum. The profit is affected by buying in larger quantities, which tends to reduce the purchase price, but increases the storage cost and the capital expenditure.

If one item only is to be considered the solution is simple. With calculus the answer becomes a junior sized formula. If, however, two items are considered simultaneously the problem becomes more complex but can still be solved by hand by plotting on a graph the number of units of each item that can be purchased for a given capital (purchasing budget) and, also, the number of units of the items that can be stored for a yearly storage cost (storage budget). The intersection of these two lines gives the most profitable combination of the two items.

For three items, the most profitable solution is indicated by the intersection of plane surfaces on a three dimensional plot. Beyond this point the solution must be done by linear programing because $n$-dimensional space would be required to extend the plotting method further.



P. H. Glatfelter Co., Spring Grove, Penna.

# How 5 grade changes a day make Glatfelter more competitive

Flexibility is synonymous with Glatfelter's No. 7 188″ machine. Their ability to make up to 5 changeovers ranging from 13 lb. bond to 110 lb. drinking cup stock means orders are shipped within hours, overruns are unnecessary and warehousing is eliminated. Rice Barton engineering has provided this versatility which makes short runs profitable and improves the mill's competitive position.

Overcoming unusual papermaking requirements with specialty machines of the most modern design has shown Rice Barton's ability as a leading manufacturer. Consult us when planning your next modernization or expansion.

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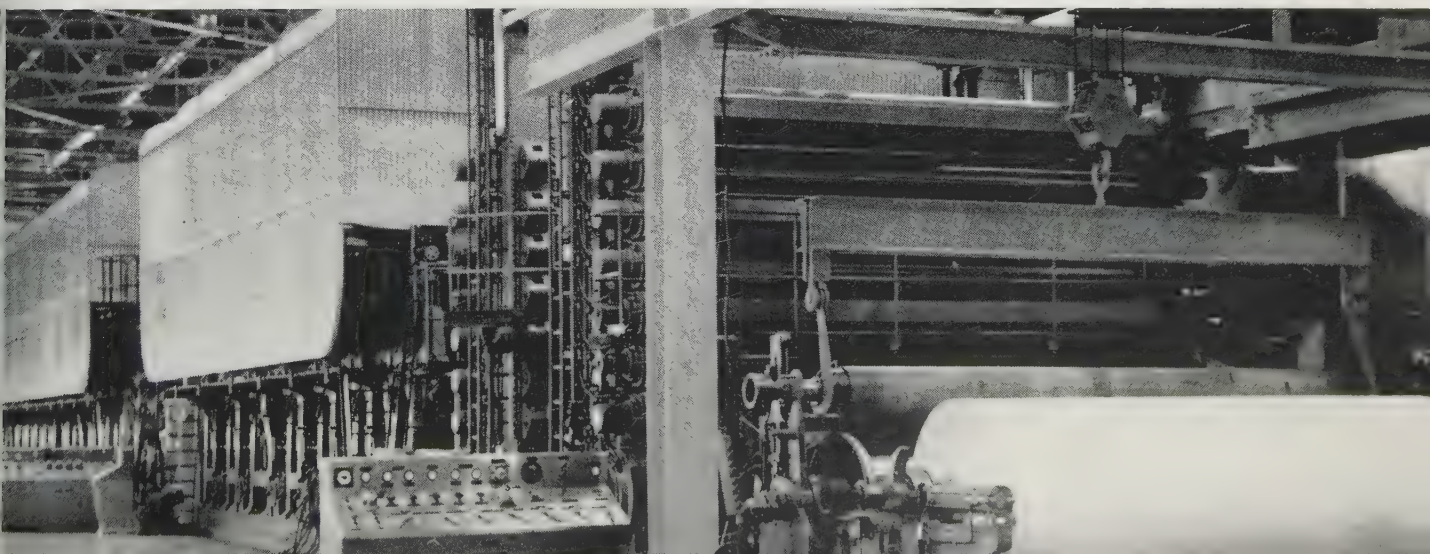

If the number of items that must be considered at one time is not large, the solution of the linear equations can still be done by hand, but in the normal case, the hand solution might require years to complete as problems have required as much as 40 hr. on a high speed computer.

Machine and building design may be of relatively small concern to the paper mill manager as these things are usually purchased under contract. But why not buy a better machine or building that was designed on a computer and had the advantages of accuracy and redesign economy possible with an electronic computer?

Problem simulation is another important and growing field of computer use. The general method of attack in this method is to construct a mathematical model of the operation to be explored and use random selection or probabilities to control unknown quantities.

Of course some information must be known about the input quantities. Past history may provide this information, or, lacking this, reasonable estimates or predictions may suffice. The main use of the computer is to run through thousands of solutions under different rules or with varying data to establish trends or patterns in the results produced.

To illustrate the use of a mathematical model let us explore the bottlenecks in the inspection, handling, cutting, and packaging of writing paper. As each order is received the operations that are required to produce the item could be written on cards. By applying past experience as a guide, the time the order would take to reach each operation and the length of time of the operation could be added to the cards. If the cards are now sorted by operations and times, the number of orders arriving at the same time at any operation becomes apparent. The bottlenecks uncovered by this model can be corrected by rescheduling some jobs. However, to do this new cards must be prepared and the whole process repeated.

This example has been oversimplified to indicate the method. Many other variables such as waiting times, machine breakdowns, etc., must be added to make this model actually useful.

In adding a variable such as machine breakdowns, random selection based on past experience would be used to determine when machines would break down and for how long they would be out of production.

This method is not practical by hand calculation because of the staggering amount of labor involved.

Perhaps the most important and lucrative field for computer use is that of production machine control, quality control and machine simulation.

This field is a comparatively new one, but progress is being made in several industries. The oil industry is perhaps further ahead than any other followed closely by the gas distribution and steel industries. Preliminary work has also been done in the cement and paper industries.

The general approach to this problem is the same for all industries since the problem is to construct a mathematical model of the production process to be simulated. In all cases the process is too involved for a direct mathematical solution so that it is necessary to rely on empirical formulas developed from data collected from the production machine.

These data, at first, may be gathered and cataloged by hand but should be, and usually are, turned over to a computer in the early stages of development because it is a tedious and time consuming chore. The computer, provided with a suitable program, is well equipped to handle the vast amount of information which must be obtained and to arrange it in the proper fashion for later use.

As most production machine instruments are of an analogy type, a converter must be obtained to transform the analogy readings to the digital input required by the computer. These devices are now made by many computer companies and can be obtained either by purchase or rental. The converters are usually capable of checking several hundred different inputs at a rate of several readings a second. This information can then be analyzed and recorded by the computer program.

From the data gathered by the computer, empirical formulas are developed to represent the action of various sections or operations of the production machine. The computer can again be of use with its various curve fit and regression analysis programs. The equations are now linked together to form the mathematical representation of the production machine and a computer program written to solve for the needed solutions.

Since this model has been constructed from empirical formulas and is undoubtedly very complex, it must be checked to determine if it is a true representation of the machine. This is usually done by connecting the computer to the production machine through its analogy to digital converter for input data and allowing the computer to type out operating instructions while the production machine is still under manual operator control. In this way the program may be debugged and checked to make certain that it is actually a true representation.

The next step, of course, is to hook up computer controls so that the computer itself can make the necessary adjustments and control the production machine itself. The program will at this time only type out such control information as is necessary to keep records and accounts on the operations performed.

Since the computer is checking at more frequent intervals and to a more precise degree than a human operator could, quality standards can be increased and downtime decreased.

In addition to these benefits, the mathematical model of the process can be used to experiment with new products or with different variations or weights of the usual product. This experimental work can be carried on rapidly and inexpensively as no equipment changes need be made nor any substandard product produced and scrapped. It is also possible to determine which parts of the production machine may be inadequate for the new product so that changes can be made before production runs are attempted.

The tasks that an electronic computer is capable of performing seem endless even though its capabilities have not yet been fully developed. As one programer has said "I don't know if a computer can ever equal human intelligence but I'm sure it will surpass it." Because of the versatility of the electronic computer it may be that man has already constructed the embryonic prototype of his most useful helper, and that the impact of this device on future social and economic conditions will lead future historians to mark this century not for its development of antibiotics, or rockets and satellites, or even the atomic bomb; but rather for the internally programed electronic computer.

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For those who wish to delve further into the world of electronic computers, the reference material listed below will furnish valuable background and information:


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Received Aug. 22, 1961. Presented at the 16th Engineering Conference of the Technical Association of the Pulp and Paper Industry, held in Washington, D. C., Oct. 15–19, 1961.

itors watch miniature Four-
nier in operation at newly
dernized Du Pont paper lab.

# EMODELED ECH LAB NOW 41 EARS YOUNG

EPWATER POINT, N. J.—A major renovation and dernization project, under way for two years and costing re than $1,500,000, has been completed at the Technical oratory of Du Pont's Dyes and Chemicals Division.

The remodeled, 41-year-old technical sales service and elopment laboratory is actually a new laboratory on the ie site. All that remains of the old building are the ls, the interior having been rebuilt entirely and re-ipped. The renovation not only provides the most dern laboratory facilities for application research in the pany's original area of dyes, but also answers the need long-range research and development of industrial micals, many of which have been introduced within last decade.

In addition to the remodeling and rearranging of space more efficient use, the newest and most advanced laboratory equipment has been installed in each section. Among the more unusual pieces are a tristimulus differential colorimeter, and a colorant mixture computer, both used in the scientific measurement of colors. All installations have been designed for maximum safety.

The Technical Laboratory has a staff of approximately 220 including about 80 who are technical experts in specialized fields of dyes and chemical applications. Besides research and sales service activities involving work on customers' problems, the laboratory maintains quality control over the plant's dyes and chemicals production, produces all the division's technical literature and serves as a training center for sales and technical personnel of both the Dyes and Chemicals Division and its customers. Laboratory representatives in an average year also make about 700 trade calls and demonstrations in customer plants.

# news from Du Pont

## REDUCE "PICK" ON PRESS, K "ZORON"* WSP FOR COATING PAPER

et lithography is based on the indisputable fact that oil water don't mix. But unfortunately the water picked by the print roll and transferred to the offset roll does with paper. Unless the paper coating is water resistant, npleasant effect called "pick" occurs, to the displeasure ie printer and his client.

Du Pont has a product—"Zoron" WSP resin finish—gned to combat this condition. Added to starch in the press and suitably catalyzed, it insolubilizes the starch boosts the water resistance of the resultant surface g. How big a boost? We happen to have some figures t here, supplied by our laboratory people who figured eone might ask:

| Paper Stock | IGT Printability Tester (feet-per-minute required to rupture paper)† | Dennison Wax Values |
|---|---|---|
| 70# offset paper, unsized | 80 | 5 |
| Same paper, sized with starch | 280 | 8 |
| Same paper, sized with starch and "Zoron" WSP | 400 | 12 |

†50 kilograms pressure, B scale, using No. 5 ink

The above salutary result, incidentally, was achieved with 20 per cent "Zoron" WSP based on the dry weight of the starch.

The significant point for papermakers is that the use of "Zoron" WSP in coating offset grades gives a paper that is more printable, has less tendency to "pick," and can be printed at higher speed—all valuable selling points for your paper.

*Reg. Trademark of E. I. du Pont de Nemours & Co. (Inc.), Wilmington 98, Delaware

## PAPER LAB TESTS ANSWER QUESTION: WHICH DYES WORK WELL WITH ALKALINE SIZE?

The Du Pont Paper Laboratory recently completed a series of beater dyeing tests comparing tinctorial strength achieved with an alkaline size versus regular rosin size and alum. The dyes tested were those normally used in the paper industry.

The complete report classifies dyes under three headings:

1. Those which have greater strength with alkaline coating than with normal quantities of rosin size and alum.
2. Those which show the same degree of tinctorial strength with either sizing material.
3. Those which show less retention in the presence of the alkaline size and should be considered unsatisfactory for use with neutral or alkaline sizing material.

In general, the tests showed that direct dyes are suitable for use with the newer synthetic size while acid dyes are not. Basic dyes color unbleached pulp satisfactorily in the presence of the alkaline size. Pigment colors in general are not satisfactory except in light shades.

For a full report on this investigation, ask your Du Pont salesman or the Dyes and Chemicals office nearest you.

## DID YOU KNOW?

. . . Wolley Segap is getting Wolley-er in many parts of country? To make their directory Yellow Pages more distinctive, some local telephone companies specifying a yellower paper than has been used in past.

. . . Speaking of yellow, the State of Kansas commem tive 4-cent stamp is printed on yellow paper? flower yellow, naturally.

. . . The country's leading graphic designers are becom even greater specifiers of paper than they have bee the past? A recent Du Pont survey among winner a national graphics contest revealed that three-quar of them are specifying paper an average of twic often as they did three years ago.

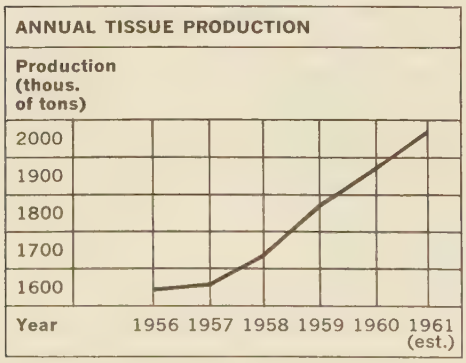
. . . Wisconsin today is the number one paper produ The Badger State turns out 10 per cent of all the p paper and paperboard made in the country.

# news from Du Pont

## TISSUE ON A TEAR

Draw a virtually straight line with a steady upward inclination and you have a graphic answer to the question of what's happening to sanitary tissue. Its production is growing at a healthy average rate of five per cent a year.

On the upgrade, too, is the proportion of tissue production devoted to color. The introduction of this "style" note into the marketing picture has permitted the housewife to indulge her natural preference for color in tissue products for the dining table, the kitchen and the bathroom.




Figures courtesy of Tissue Association

Which brings us inexorably to the fact that Du Pont has a lineup of dyes excellently suited to the requirements of tissue mills. "Pontamine"® Bond Yellow CS, for example, is ideal for yellow shades. "Pontamine" Fast Scarlet 4BA and "Pontamine" Fast Red 8BLX are the best colors for pink tissue on the market, distinctly superior in working qualities.

Listed below are a broad range of Du Pont dyes for tissue. Talk to your Du Pont salesman about the whole line. It's a favorite topic with him.

**TISSUE COLOR TABLE**

| Shade | Suggested Du Pont Color |
|---|---|
| Yellow | "Pontamine" Bond Yellow CS<br>"Pontamine" Fast Yellow 4GL<br>"Pontamine" Yellow CHP |
| Orange | "Pontamine" Fast Orange WS<br>"Pontamine" Fast Orange 6RN |
| Pink | "Pontamine" Fast Red 8BLX<br>"Pontamine" Fast Scarlet 4BA<br>"Pontamine" Fast Scarlet 4BSP |
| Brown | "Pontamine" Fast Brown NP<br>"Pontamine" Fast Brown RMR |
| Blue | "Pontamine" Sky Blue 6BX<br>"Pontamine" Bond Blue B<br>"Pontamine" Fast Turquoise 8GLP |
| Green | "Pontamine" Green BXPW<br>"Pontamine" Sky Blue 6BX<br>"Pontamine" Bond Yellow CS |

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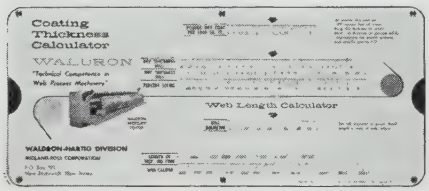
A full range of standard 3 roll and 4 roll designs from "pilot plant" size to 154" widths are available. Write for specifications.

Laboratory facilities are also available for your use in evaluating Waldron equipment.

*Write for new convenient "Coating Weight Calculator". No obligation.*

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***NEW BECCO SINGLE-STAGE SYSTEM OFFERS HIGHER PULP BRIGHTNESS AT LOWER PEROXIDE COSTS***

Since March 1960, St. Regis Paper Company has been bleaching groundwood pulp at a 25% consistency with a new single-stage bleaching process developed by Becco. Hydrogen peroxide bleaching solutions are consistently yielding brightness levels of 75-78 from unbleached pulp fed into the system at a brightness level of 60.

In the St. Regis, Deferiet, N. Y., operation a newly designed vertical screw press (developed by FMC Corporation's Canning Machinery Division) dewaters the pulp, permitting the most efficient use of the peroxide.

Full details of the St. Regis operation were presented in Chicago in a joint report by St. Regis and FMC at the T. A. P. P. I. meetings September 19-20-21.

The results at St. Regis, Deferiet, clearly demonstrate the success of commercial high-density groundwood bleaching. If you are looking for higher brightness in your groundwood bleaching operation, contact Becco for details on this proven system. Address your inquiries to: Dept. T-61-23; Becco Chemical Division, FMC Corporation, 161 East 42nd Street, New York 17, New York.

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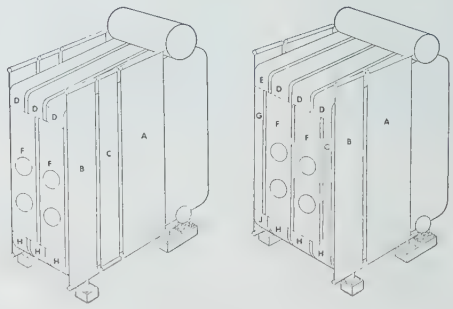
**FMC CORPORATION**

*The New C-E VU-60*

# MODULAR BOILER

(100,000 to 250,000 lb/hr)

The modular construction of C-E's new VU-60 makes it possible to assemble custom boiler designs from standardized components. Boilers can be proportioned for efficient combustion, heat absorption and gas flow – and conform to wide variations in space conditions.



*These dimensions . . . . or these give you the same capacity and efficiency*

Letters on drawing represent a few of the standardized VU-60 components (or modules) from which – for a given capacity requirement – a wide range of boiler proportions can be obtained.

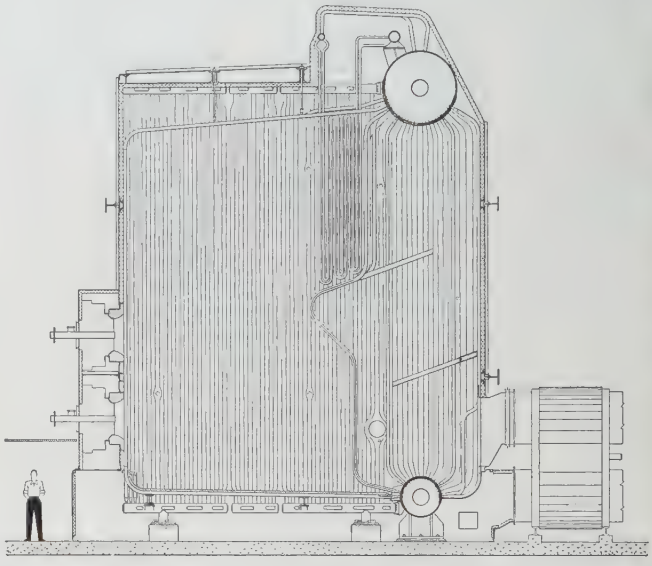
## Self-Cased

The VU-60 is *completely* self-cased. At the shop, the fins of furnace and boiler tubes are welded together to form panels. These sections are then field-welded into a pressure-tight unit that offers a minimum of weight per pound of steam capacity.

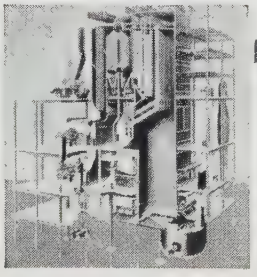
## Simplified Design

Economical, dependable and accessible, the VU-60 has no external ductwork. Installation is made on a simple, reinforced concrete slab – the bottom of the boiler forming a plenum. Cross-flow baffling and symmetrical boiler bank minimize draft loss. For oil or gas firing.

You can depend on this economical, standardized boiler for the efficient production of clean, dry steam . . . for power or processing.



WRITE FOR LITERATURE



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**MORE THAN** paper is thrown away when the efficiency of the size has not been fully realized. Time and money are also wasted. Don't let your mill face these losses. Consider Cyanamid's **high quality rosin sizes** — fortified and unfortified, liquid and dry — for dependable, cost-saving performance. And in back of Cyanamid's **high quality rosin sizes** are those all-important hidden ingredients... Cyanamid's extensive manufacturing and research facilities, and the skilled personnel who man them. Your Cyanamid salesman, with his technical training and paper-making knowledge, is truly your partner in paper progress. Ask him to give you complete information about Cyanamid's **high quality rosin sizes.**

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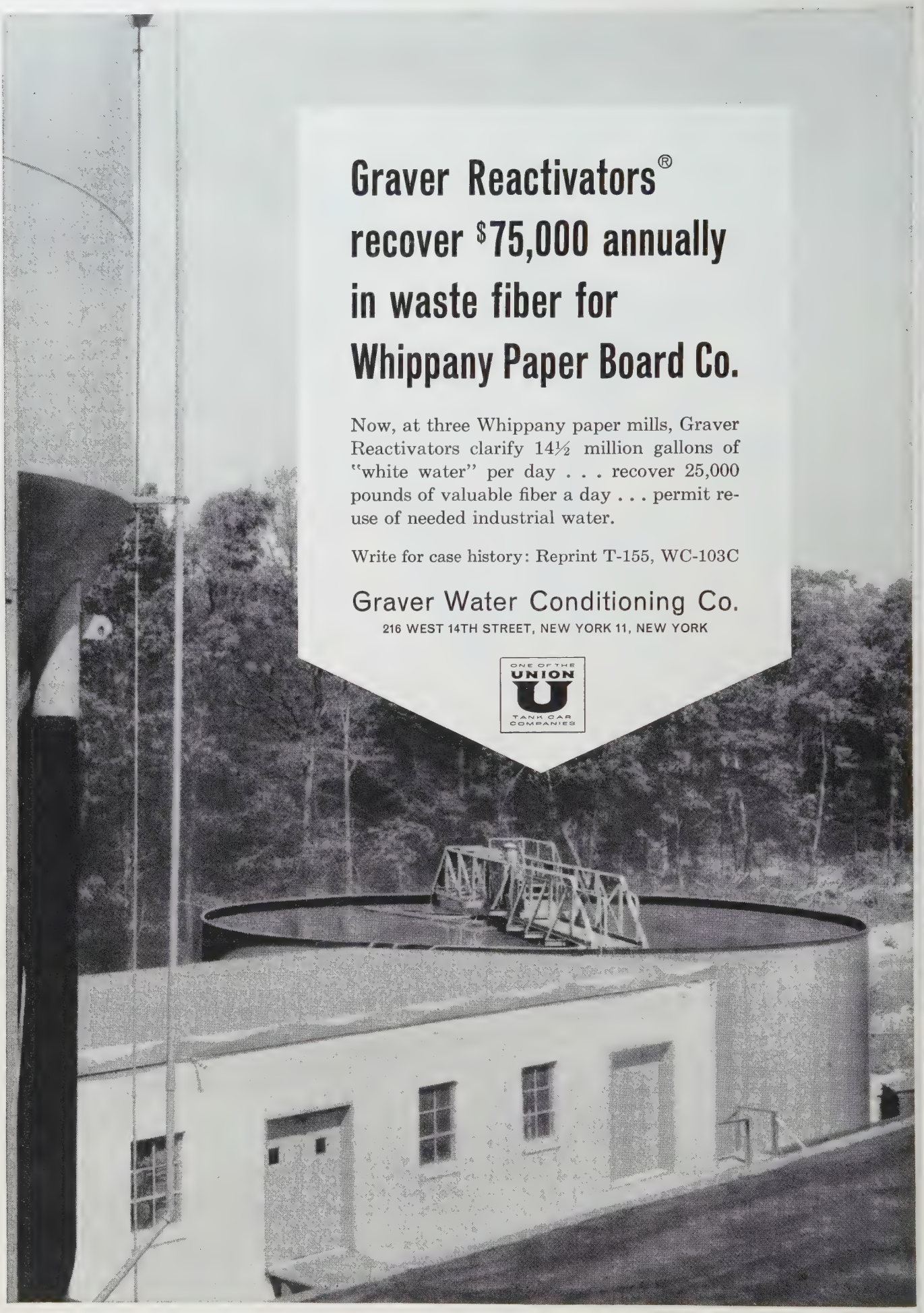

# Graver Reactivators® recover $75,000 annually in waste fiber for Whippany Paper Board Co.

Now, at three Whippany paper mills, Graver Reactivators clarify 14½ million gallons of "white water" per day . . . recover 25,000 pounds of valuable fiber a day . . . permit re-use of needed industrial water.

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## When You Use

# Nalco® 680 SP

## Sodium Aluminate

### A QUALITY RAW MATERIAL FOR:

**Increased**

- ...Permanence
- ...Strength
- ...Retention

**Improved**

- ...Formation
- ...Sizing
- ...Paper Machine Operation

One of the many ways in which quality is measured in Nalco laboratories—paper chemist Glenn Goldsmith determines the strength of a paper sample with a tensile tester.

**Nalco 680SP Papermakers' Sodium Aluminate improves paper quality — and reduces size and alum requirements, providing:**

Increased Permanence—Proper use of Nalco 680SP reduces excess acidity and sulfate ion concentration in the furnish. Since sulfates hydrolyze during storage to produce paper-degrading sulfuric acid, a reduction in sulfates and excess acidity will result in paper with superior aging qualities.

Increased Strength—Proper use of Nalco 680SP in the furnish results in stronger paper, for several reasons. Retention and formation are substantially improved, making a stronger sheet. Nalco 680SP also reduces dissolved solids, resulting in better fibre-to-fibre bonding.

Increased Retention—Nalco 680SP, an excellent flocculant, forms a uniform alumina floc which retains more filler, fines, size, and other furnish constituents in the sheet. *More* of the furnish becomes salable paper.

Improved Formation—Increased retention, improved bonding, and the uniformity of the sodium aluminate-alum floc greatly reduce clumping, which causes poor formation. Two-sidedness and "wild" formation are minimized.

Improved Sizing—Nalco 680SP combines with alum to provide an optimum alumina/size ratio at the most desirable pH for size precipitation. At the same time, there is a substantial reduction in sulfate ion concentration, soluble alumina, and excess acidity, all of which are detrimental to proper sizing.

Improved Machine Operation—More effective retention of fines and fillers, the reduction of dissolved solids, and the completeness of the 680SP-alum-size reaction all combine to reduce deposit formation and plugging of wires, felts, suction boxes, and couch rolls. The result is a cleaner machine system, free of chemical slime. In addition, reduced acidity and increased pH result in sharply reduced corrosion rates.

### Nalco 680SP: A Chemical That Brings Results

Results with Nalco Sodium Aluminate—and every other Nalco chemical used in paper processing—can be demonstrated by Nalco Representatives. Their experience and willingness to help can be of real value to you . . . whether the situation calls for emergency action or long-range planning for better paper products at lowest possible production costs.

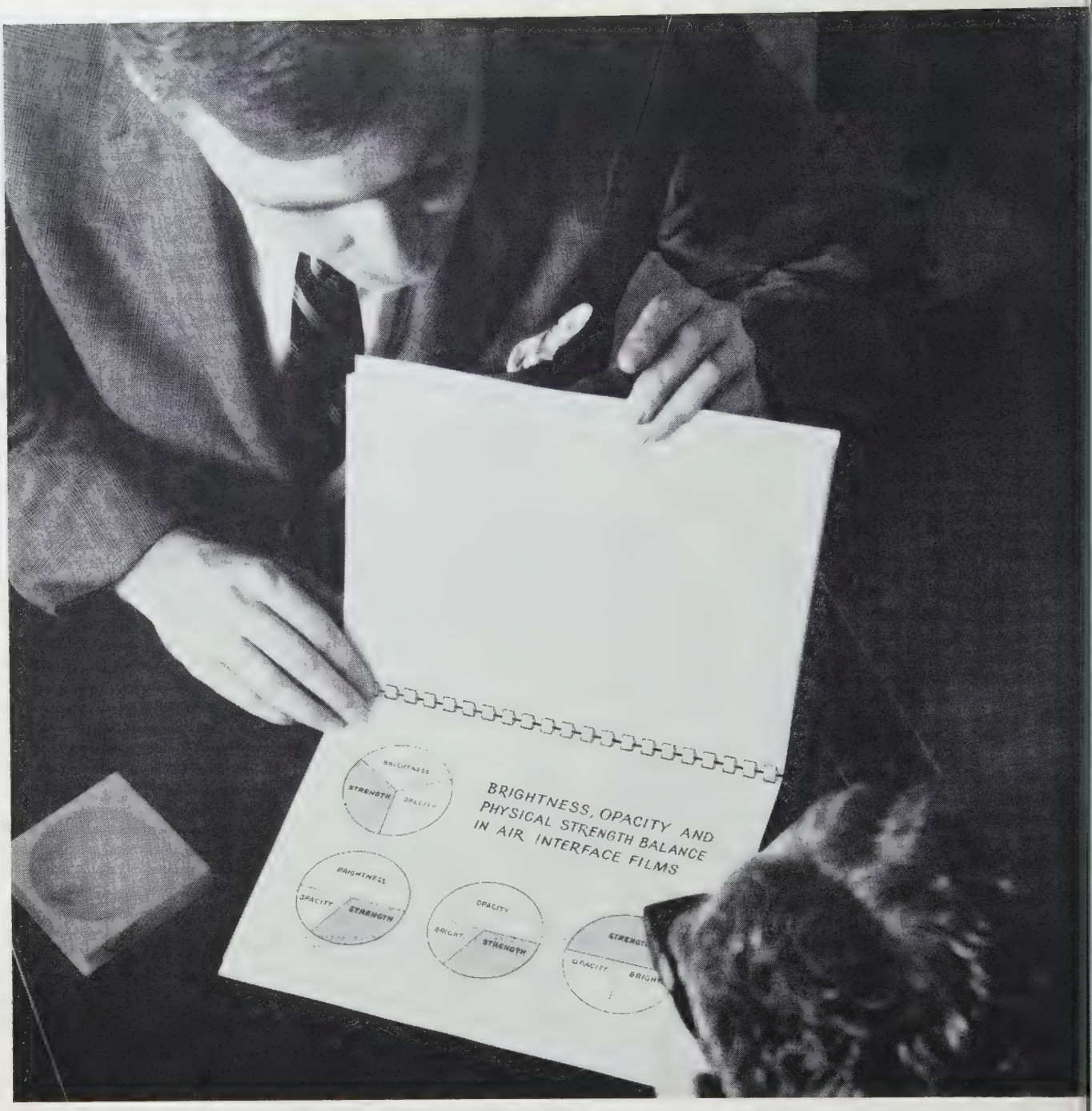
For action that leads to results like these in your mill, call your Nalco Representative, or write:


**NALCO CHEMICAL COMPANY**

6197 West 66th Place • Chicago 38, Illinois

*Subsidiaries in England, Italy, Mexico, Spain, Venezuela and West Germany*
*In Canada: Alchem Limited, Burlington, Ontario*


Nalco® ...Serving the Paper Industry through Practical Applied Science




# Let Du Pont help you get the right opacity at the right price

When you buy white pigments, you're buying opacity. But if your choice of pigments gives more opacity than you need or opacity at a sacrifice of brightness and strength—you're wasting money!

And here is where your Du Pont Pigments Representative can help you. He can draw upon the widest choice of pigments available to give you just the opacity you require, while maintaining all other desirable properties. Du Pont's broad line of "Ti-Pure" titanium dioxide pigments includes many new grades not available elsewhere—grades that were developed especially for the paper industr

Call your Du Pont Pigments Representative. He prove to you the important thing is not what yc pay for the pigment—it's what you pay for opacit

Du Pont, Pigments Dept., Room D-2060, Wilmington 98, Delawa
In Canada: Du Pont of Canada, Ltd., P.O. Box 660, Montreal, Queb

DU PONT REG. U.S. PAT. OFF. **TI-PURE®** titanium dioxide pigments

BETTER THINGS FOR BETTER LIVING...*THROUGH CHEMISTR*

BIRD & SON, INC.
East Walpole, Mass.

# One hundred and sixty-six years ago...

In the year 1795 George Bird moved from Maine to Massachusetts and started a small paper mill. Today, Bird & Son, Inc., has expanded into a multi-product manufacturer and its Paper Division makes everything from shipping cases to tack boxes. For the past ten years they have been using Cities Service Pacemaker 35 Wax on all their waxed cartons.

The Bird Company finds that Cities Service Pacemaker Wax gives complete coverage to reduce waste . . . seals hard for increased durability . . . makes the finished product a real work of art . . . a rugged one, too.

Whatever your wax needs, you'll find that a Cities Service Pacemaker wax can do the job better. And you can rely on Cities Service to provide expert technical help . . . fast, dependable service, too. For more information, call your nearest Cities Service office or write: Cities Service Oil Company, 60 Wall Street, New York 5, N.Y.

CITIES SERVICE

QUALITY PETROLEUM PRODUCTS

*How a groundwood mill screens knots and slivers automatically with the*

# DORRCO® BAR SCREEN

At Consolidated Paper Corporation, Grand'Mere Quebec, removal of long wood slivers and knots from raw groundwood stock was previously accomplished by a combination of screening through a bar rack and manual raking.

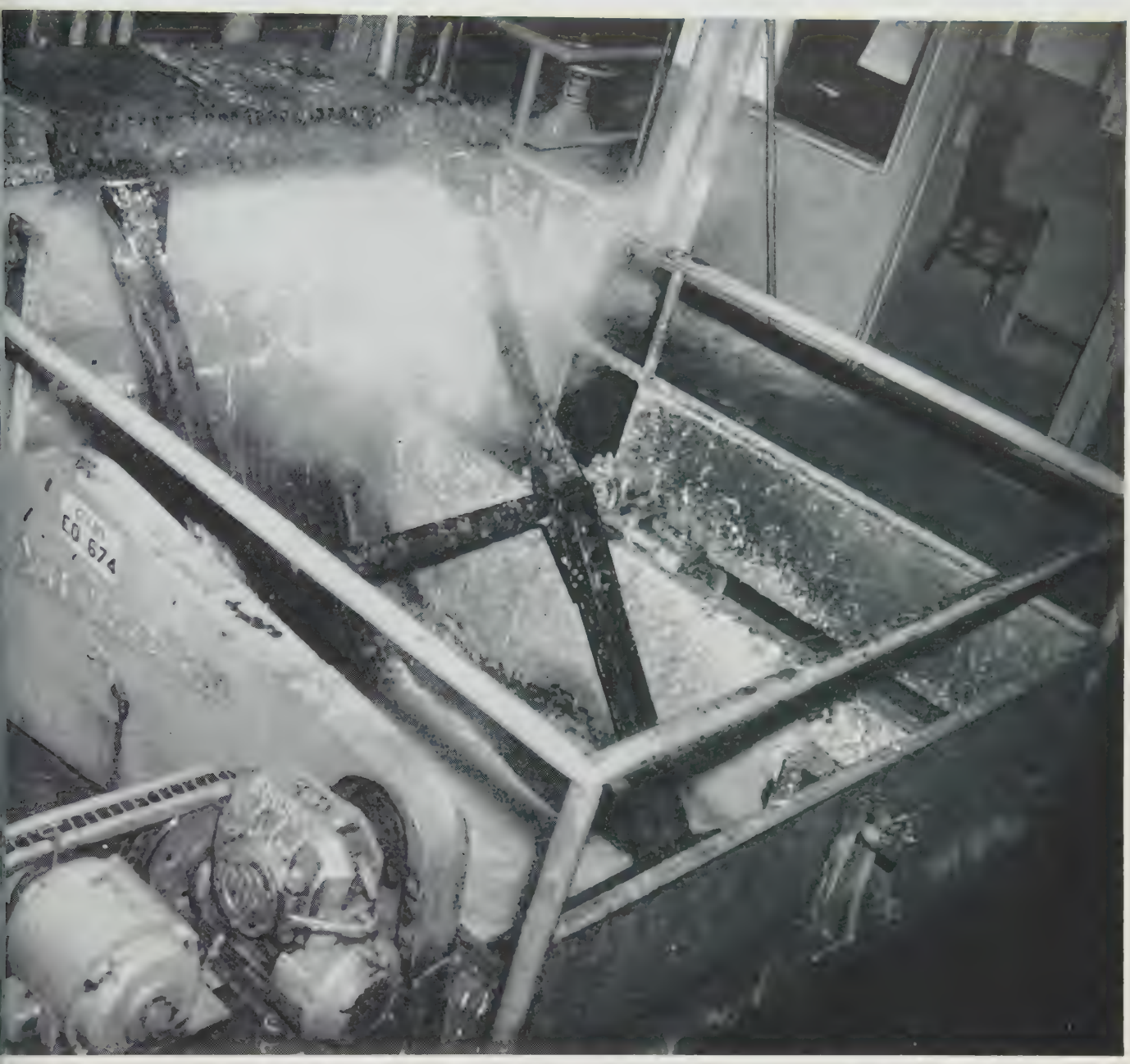
The job is now done automatically and with considerable saving in overhead costs by the unique equipment shown in the photograph. It consists of a bar screen shaped to form a quadrant and continuously cleaned of screened material by a revolving rake mechanism. This unit is installed in a channel 5′6″ wide and 3′ deep. It handles approximately 9,000 gpm of 1% consistency groundwood stock. Screenings comprise about 15 cu. ft. per million gallons.

The Dorrco Bar Screen was originally produced for sewage and waste screening. Several hundreds of successful installations for this purpose are in operation. The equipment is simple, rugged and requires little power. A variety of sizes are available and in many cases can be fitted into existing channels. The Consolidated Paper Corporation installation represents the first adaptation of the unit, which was modified and strengthened for use in the pulp and paper field. Opportunities exist for wider application.

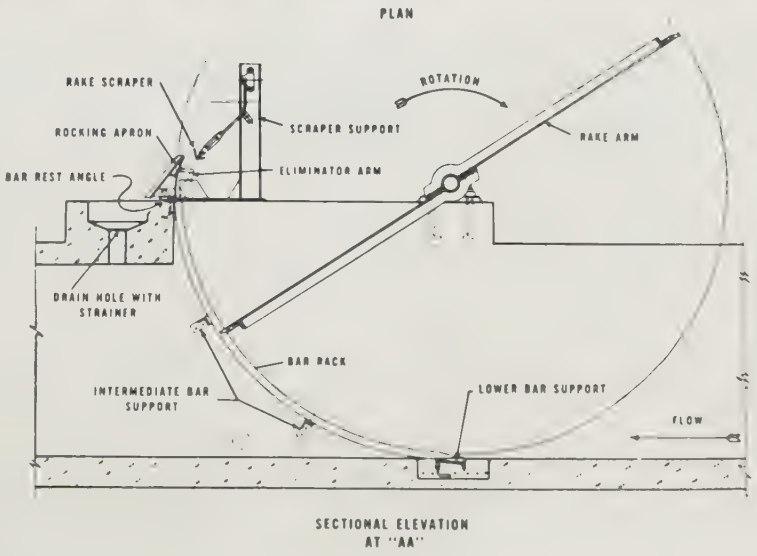
For full information, write to Dorr-Oliver Incorporated, Stamford, Conn.

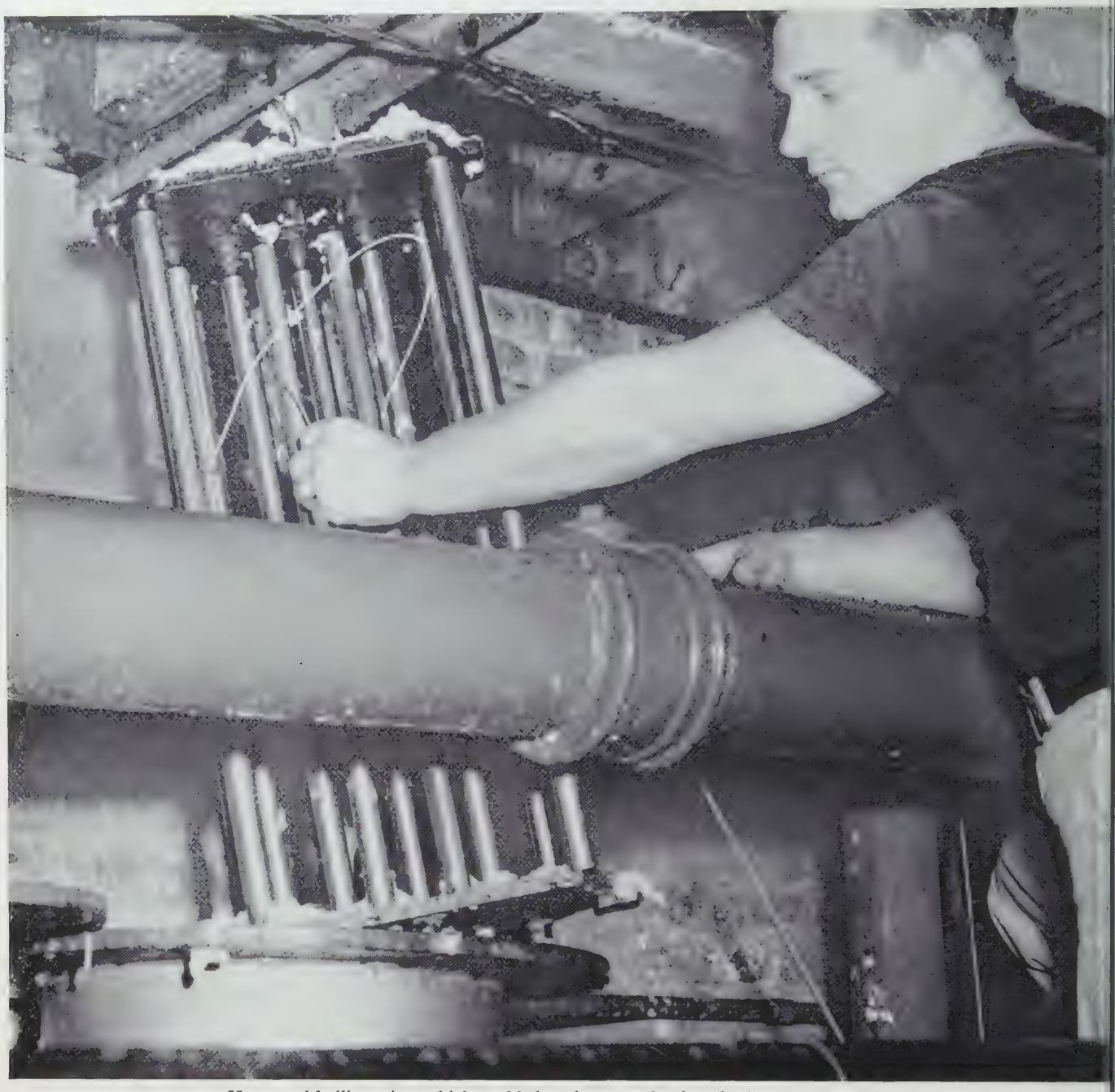
DORR-OLIVER

WORLD-WIDE RESEARCH • ENGINEERING • EQUIPMENT



Operation of the Dorrco Bar Screen is demonstrated in the photograph and the drawing at right. Flow is against the concave side of the screen. Plates at the ends of the revolving rake are toothed to dovetail into the bar openings, preventing any possibility of clogging. Screenings are continuously swept upward and then removed from the rakes by a scraper bar attached to a rocker arm.





Unwanted bailing wire—which could shut down production—is shown captured in an Eriez magnetic trap mounted in the paper slurry pipe line at Doeskin Products.

# How Nickel stainless steel sets a trap for tramp iron . . . and high costs in pulp slurry

Pictured above is a magnetic trap used to remove tramp iron from the pulp slurry at Doeskin Products, Rockland, Delaware. This iron must be removed—otherwise the flow could be brought to a costly halt.

The magnets are enclosed in Type 304 Nickel stainless steel tubes. The traps—developed by Eriez Manufacturing Company, Erie, Pennsylvania—are installed between a large and a small hydrapulper and their holding chests. Nickel stainless steel has meant considerable savings for Doeskin Products because it has stood up to the attack of urea resins and alum in the slurry. This slurry had previously destroyed iron magnets.

These magnetic traps are a good example of why you should look to Nickel stainless steel for outstanding resistance to corrosion. But you can also look to the Nickel stainless steels for other good properties *in combination* with corrosion resistance—such as resistance to abrasion, erosion, impact, as well as weldability and ease of maintenance and cleaning.

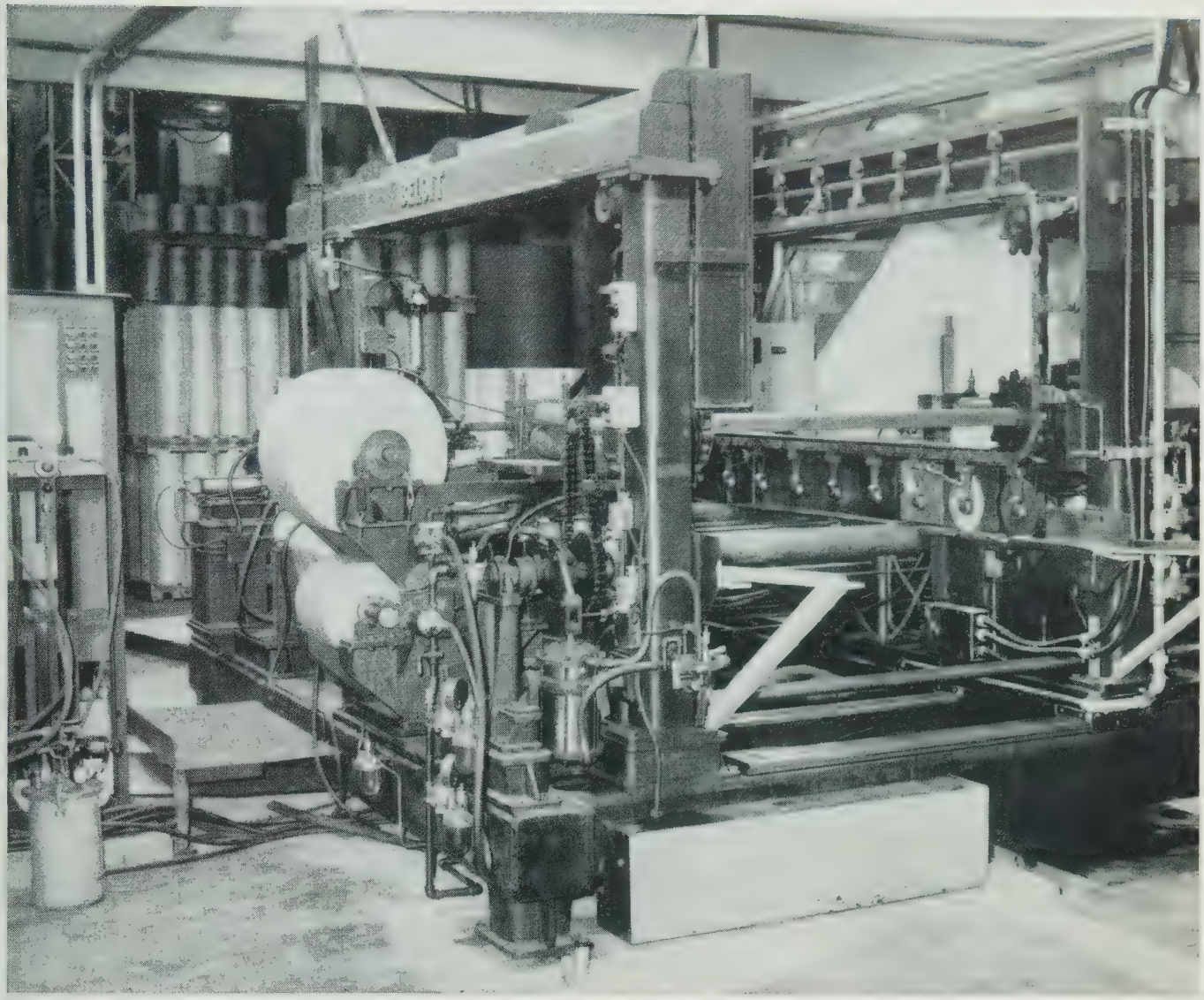
To find out just how the Nickel stainless steels can help your pulp and paper operations, write to Inco describing your design and metals needs.

THE INTERNATIONAL NICKEL COMPANY, INC.

67 Wall Street INCO New York 5, N. Y.

**INCO NICKEL**

**makes alloys perform better longer**



S. R. CHRISTENSEN

International Paper Co., Mobile, Alabama

# Beloit counter roll wrapper

Compact, well-engineered unit puts counter roll wrapping on a completely automatic basis. A Beloit counter roll wrapper can be arranged to handle counter rolls of any size. Consider these important features:

• plugs, wraps, crimps and glues automatically • wrapper sheets automatically cut from a single parent roll • end covers die cut and applied from a single parent roll • neat, uniform customer rolls • reject arrangement for nonstandard rolls • speed range of ten rolls per minute

**FINISHED PRODUCT PROCESSING EQUIPMENT**

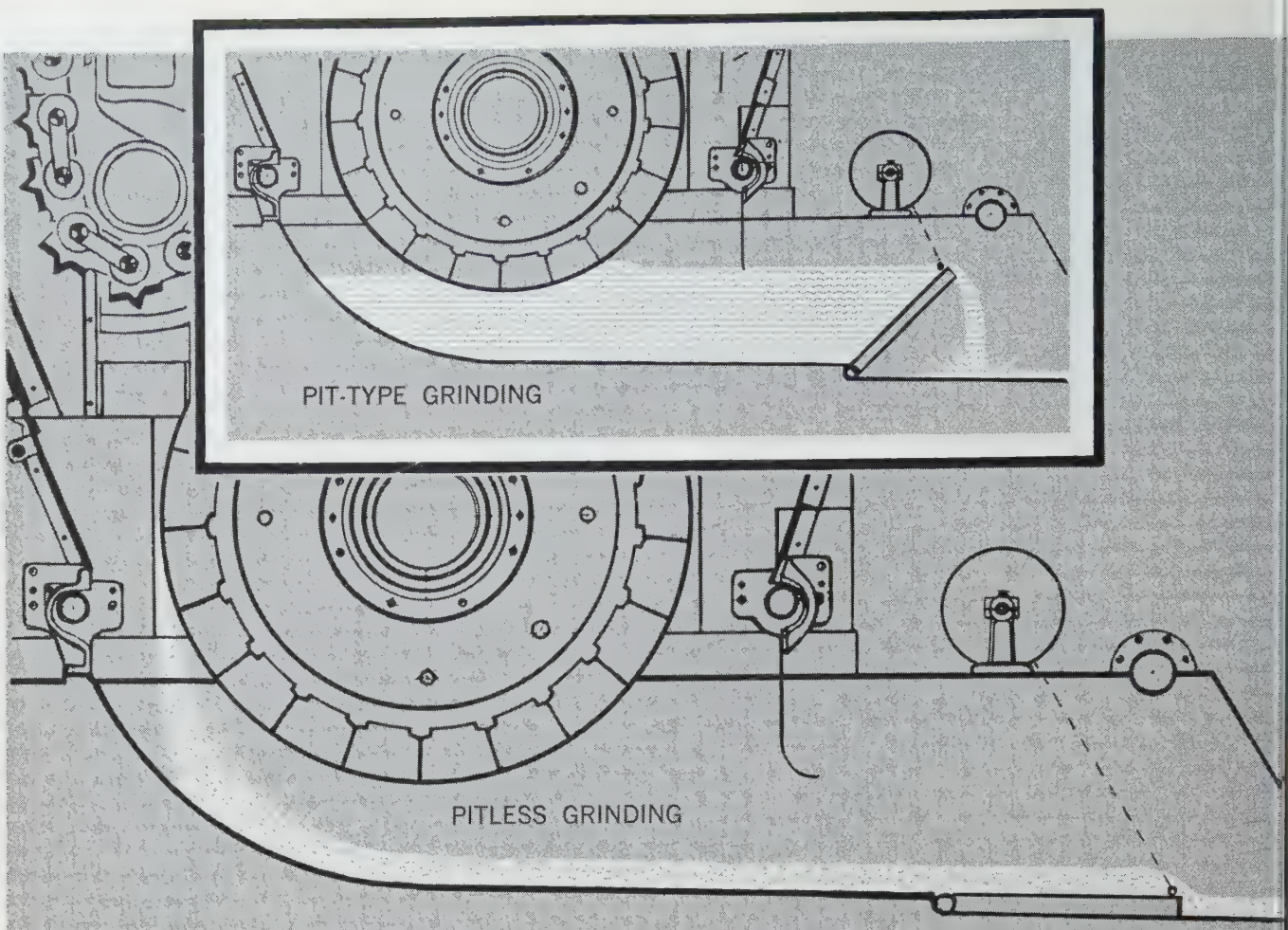
Winders • Reclaim Winders • Cutters
Sheeters • Slitters • Supercalenders
Roll Wrappers • Roll Lowering Tables
Roll and Shaft Handling Equipment
Conveyors • Unwind Stands and
Tru-Tension Controls • and other
equipment for the paper and
allied industries.

BELOIT EASTERN PAPER MACHINERY DOWNINGTOWN, PA.

Member Beloit Group

**BELOIT EASTERN CORPORATION**

DOWNINGTOWN, PENNSYLVANIA

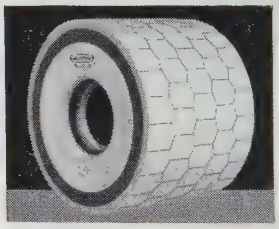



# Pitless Grinding

## IS an advantage . . .

*Mills report:*

- **10% increase in production**
- **Longer stone life**
- **Extended burring cycles**
- **More uniform pulp**



From the beginning, Norton pulpstone engineers have been associated in the development of pitless grinding. They are familiar with the technique and will show you how your present equipment can be adapted to this more efficient, more profitable method. Call Worcester, SWift 8-2511, or Hamilton, LIberty 4-3321.

NORTON COMPANY, WORCESTER 6, MASS.
Norton Company of Canada Ltd., Hamilton, Ont.
Norton International Inc., Worcester 6, Mass.

NORTON PULPSTONES — *the world's standard for mechanical pulp*

Making better products . . . to make your products bette

NORTON PRODUCTS: Abrasives • Grinding Wheels • Machine Tools • Refractories • Non-Slip Floors — BEHR-MANNING DIVISION: Coated Abrasives • Sharpening Stones • Pressure-Sensitive Ta

## RHOPLEX® helps packages ring up more sales

This advertisement is printed on standard bleached kraft folding boxboard. It shows how RHOPLEX coatings enhance the quality of many types of packages. The RHOPLEX binder in the coating gives the surface a smoothness and gloss comparable to high-quality printing paper. The result is vivid colors, and extremely precise halftone dot definition. This means maximum eye-catching power, maximum persuasiveness, maximum sales impact. Because RHOPLEX binders are flexible, they prevent the coating from breaking when boxboard is scored and folded.

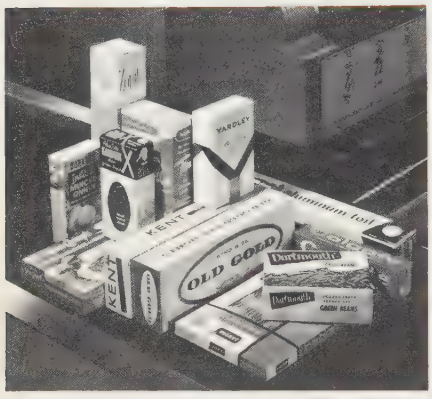
RHOPLEX binders are chemically and mechanically stable, trouble-free and easy to use on a wide variety of paper and paperboard. Write for the technical booklet "RHOPLEX Acrylic Emulsions in Paper Coatings."

ROHM & HAAS

PHILADELPHIA 5, PA.

# RHOPLEX

# How RHOPLEX® all-acrylic paper coating binders improve printing properties ...make processing easier




**Plus for package printing.** RHOPLEX binders flow and level under the heat and pressure of the calender rolls. The result is a uniformly smooth and glossy surface. Colors are brighter, printing definition better than when natural binders are used. Ink receptivity is good, and the paper surface has controlled ink hold-out, good varnish hold-out, and very great resistance to picking. Package designers can make full use of colors, illustrations, and halftones, without being limited by the possibility of poor reproduction.

**Easy application.** Good results are obtained with RHOPLEX-bound coatings in a variety of different equipment. Coatings may be applied on trailing blade coaters, air knife coaters, Champion coaters, on the size press, and on the calender stack.

**Processing advantages.** Formulations using RHOPLEX as binder are chemically and mechanically stable. Coatings containing up to 65 percent solids have good working viscosity, provided the binder is mainly RHOPLEX. In many cases, total binder content of formulations can be reduced when RHOPLEX is used, since it has unusually high bonding strength. Pigmented coatings have excellent color stability. Special grease-resistant coatings for packaging and similar applications may be readily formulated with RHOPLEX binders.

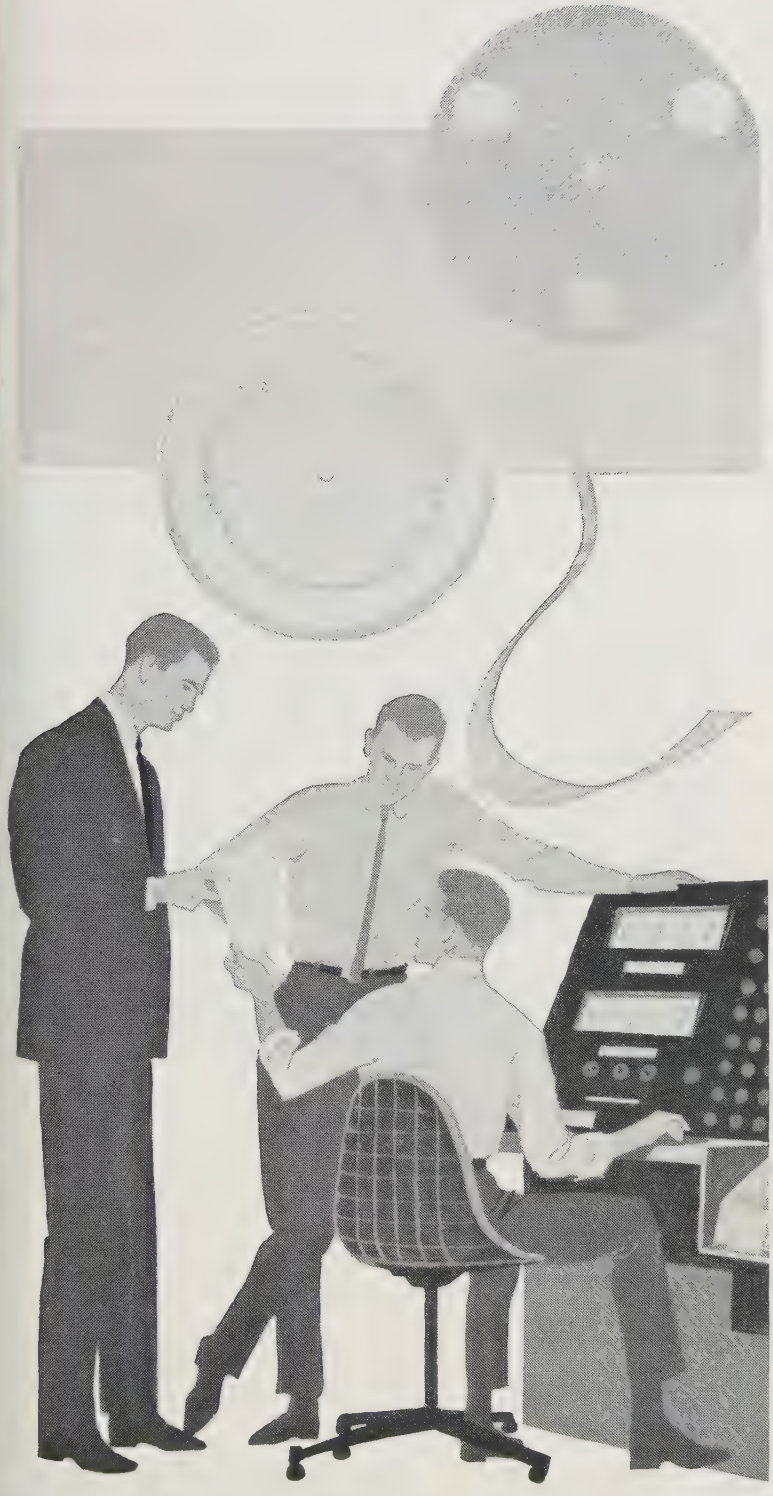
**Write for a free copy of the latest technical booklet on RHOPLEX paper coating binders.**

ROHM & HAAS
PHILADELPHIA 5, PA.

RHOPLEX

# HOW FAR . . . AND HOW FAST . . .

## can you use computer control *profitably?*



### BAILEY 700 SYSTEMS CAN HELP YOU DETERMINE THIS—ONE STEP AT A TIME

Can computer control be used—profitably—in your process operations?

Where and how?

The answer often lies in intermediate stages of improvement in sensing, measuring, controlling, computing.

Bailey engineers can help you determine and prove — one step at a time — how far analog or digital systems can be profitably applied in your operations. Bailey is equipped, by broad experience, to provide single-source responsibility from sensing and measuring instrumentation to complete automation. *Bailey 700 Systems* are installed, in operation, or on order, at locations from coast to coast and throughout the world.

Find out what this Bailey background can offer. Contact your Bailey District Office.

### SIX STEPS TO FULL AUTOMATION

**Centralize and simplify information display**—use analog and digital techniques to clarify information, aid operator understanding, improve reliability of interpretation, and save space.

**Extend use of interlocks and limiting circuitry**—use digital solid-state components to extend supervisory controls, thereby minimizing effects of human error.

**Increase use of automatic sub-loops**—simplify and standardize the starting, controlling, and stopping of major plant components and sub-systems.

**Extend on-line controls**—integrate sub-loops, interlocks, and limiting controls with conventional controls to secure automatic operation over full range, once unit is on the line.

**Provide performance monitoring**—add computing facilities to provide significant up-to-date calculations for operation improvement and maintenance scheduling.

**Integrate all system elements**—add start-stop control to provide full automation, the ultimate goal.

P 158-1

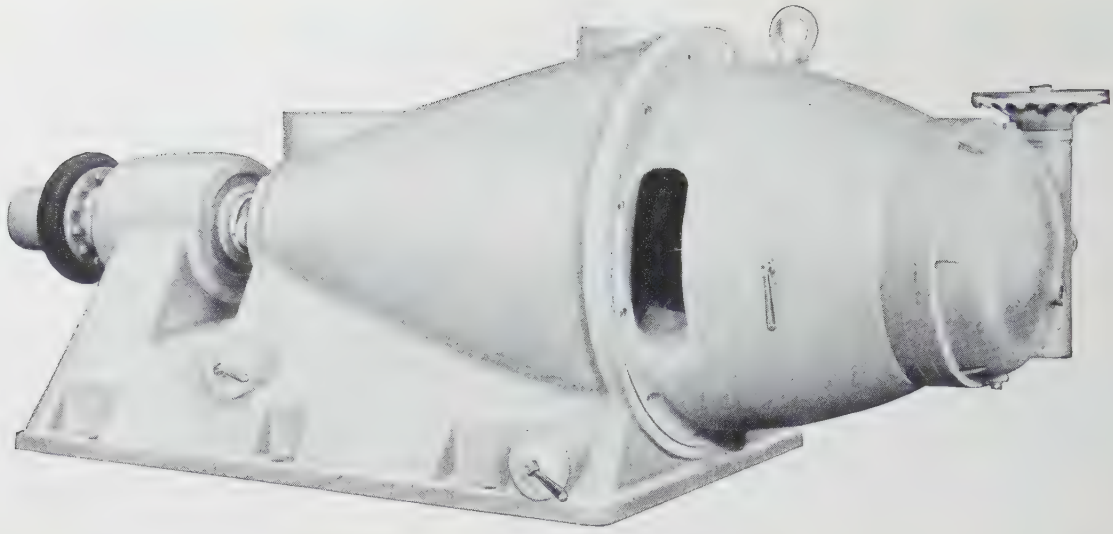
PULP AND PAPER DIVISION

**BAILEY METER COMPANY**

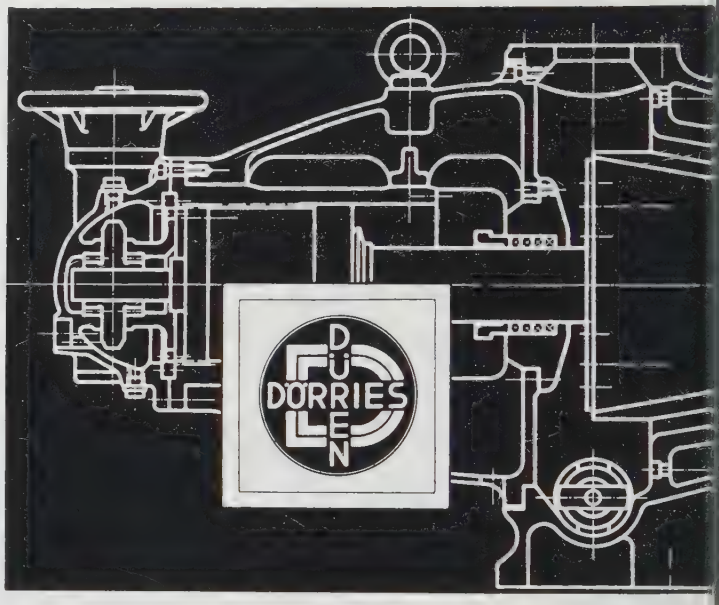
1077 IVANHOE ROAD • CLEVELAND 10, OHIO

In Canada—Bailey Meter Company Limited, Montreal

BAILEY METERS



# THE NEW DÖRRIES-REFINER




- Can be supplied in 3 sizes for a power consumption from 35 HP to 175 HP.
- Sturdy closed design.
- Refining fittings of steel casting, basaltic lava and/or special knives specifically designed for the refining process in question.
- Extra strong bearings with reliable oiling system and stationary inspection glasses.
- Exact setting precision graduated either by hand wheel or electrically.
- Please ask for our new prospectus.

## O. DÖRRIES A.G. DÜREN

EisengieBerei und Maschinenfabriken • Western Germany

**Agents: Bulkley, Dunton Pulp Company, Inc. 295 Madison Avenue, New York 17, N. Y./USA**
Telephone: MUrray Hill 9-6400 Telex: TWY-NY 1-2092

# *Only* MANHATTAN RUBBER COVERED ROLLS

## Assure ALL These Performance Advantages

- Uniform Squeeze . . . Sure Release
- No Pick-up . . . No Crush
- Longer Felt Life
- Won't Crack, Corrugate or Oxidize
- More Water Removal— at Lower Dryer Steam Costs
- High Machine Speeds . . . Lower Production Costs
- Require Minimum Regrinding—Even Under Extreme Conditions of Use

Advanced roll covering facilities and skilled craftsmanship at Manhattan make the difference. Manhattan's inseparable rubber-to-metal bond eliminates cover separation. Manhattan's grinding and vulcanizing facilities insure uniform, exact density—and accuracy of crown dimensions within .002". Manhattan's electronic balancing of rolls—at *your* machine speeds—guarantee *uniform* pressure and moisture distribution in the stock . . . less wear on felts, bearings and journals.

**GET "MORE USE PER DOLLAR" ON EVERY CRITICAL ROLL FUNCTION WITH THESE MANHATTAN RUBBER COVERED ROLLS.**

***• Press Rolls—Ray-Roc, Rub-Roc, Sham-Roc, Self-Skinner • Suction Press and Coating Rolls • Sealed-End Couch Rolls • Marblex Table Rolls***

Facilities near you . . .

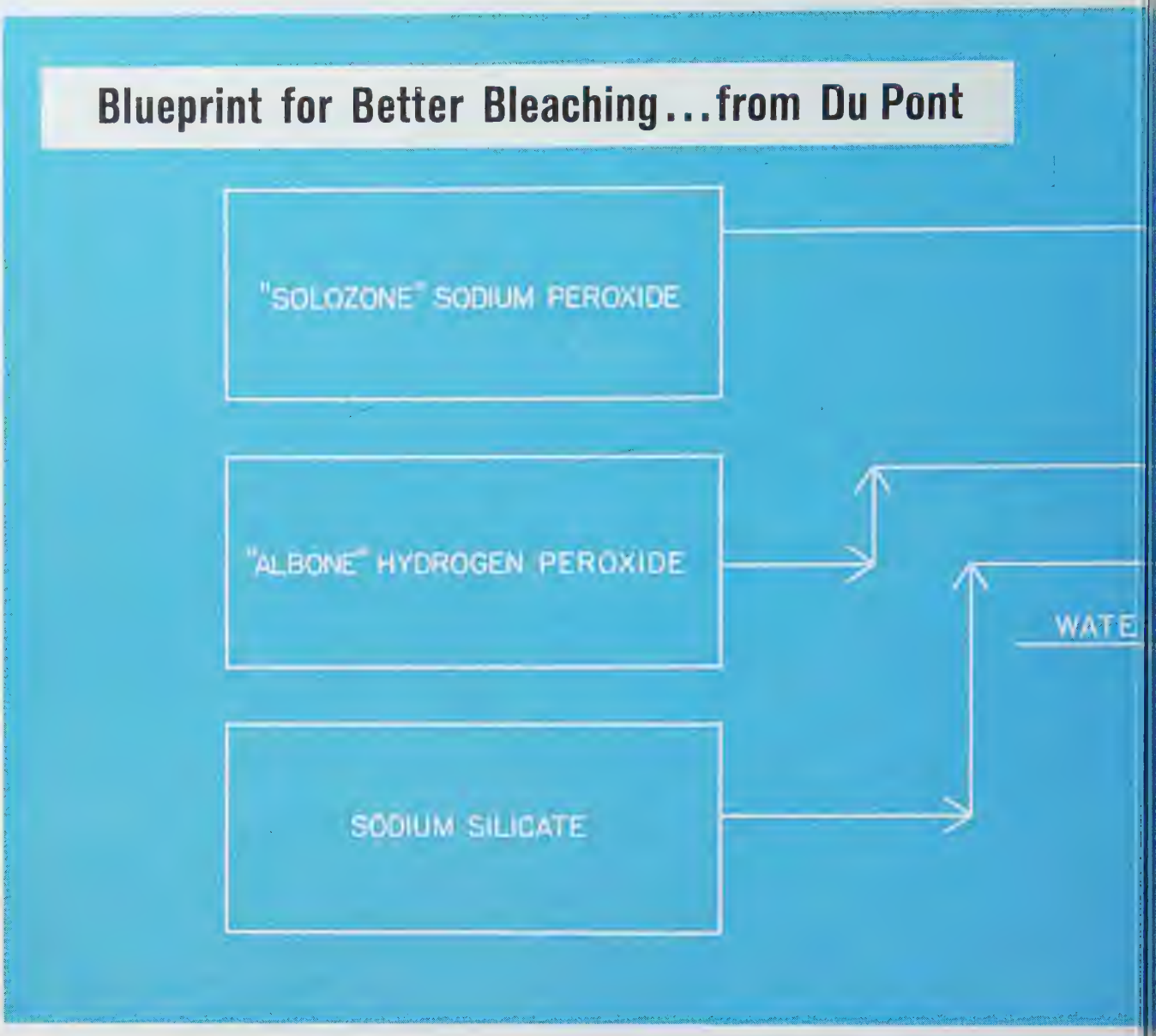
ROLL COVERING PLANTS AT PASSAIC, N.J. • NEENAH, WISC. • N. CHARLESTON, S.C.

**RAYBESTOS-MANHATTAN, INC.**

**MANHATTAN RUBBER DIVISION • PASSAIC, N. J.**

*ENGINEERED RUBBER PRODUCTS*

RM136




A typical automatic make-up system for peroxide solution using a combi

# How you can use a combination of Du Pont Peroxides

Practically all groundwood bleacheries have found that a combination of hydrogen and sodium peroxides cuts bleaching costs. The reason—combination formulas eliminate the use of caustic soda.

How is this possible? Because of the bleaching chemistry involved. When hydrogen peroxide is used alone, substantial quantities of caustic soda must be added to assure effective bleaching. But when sodium peroxide is part of the formula, caustic soda is formed and additional caustic isn't needed. Sufficient sodium peroxide is dissolved to supply the necessary caustic, hydrogen peroxide is added to complete the peroxyge balance and the bleaching formula is now ready to use.

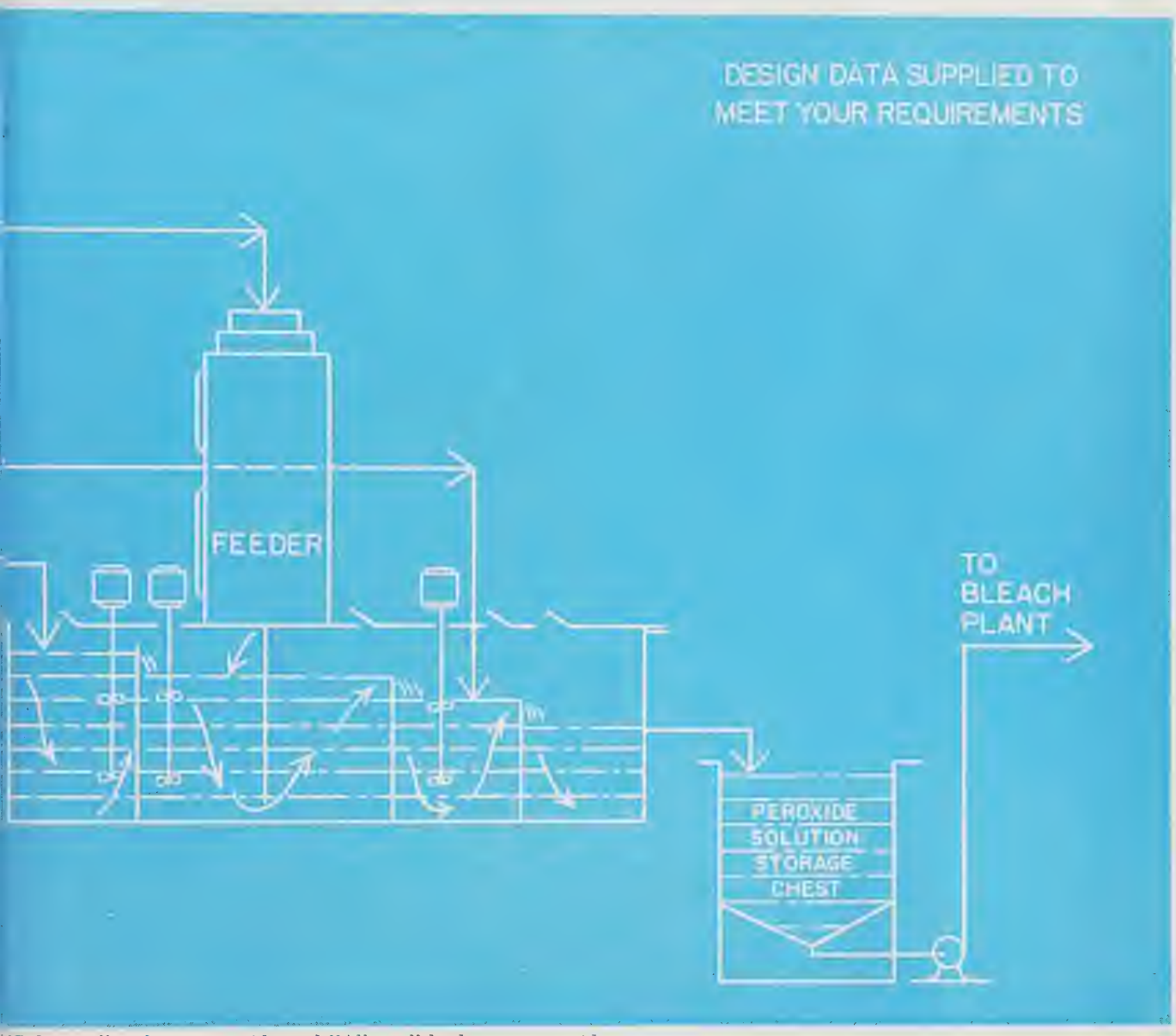
The tables show typical hydrogen peroxide formula for one-stage tower bleaching of groundwood an equivalent formulas using a combination of peroxides. Savings from use of the combination formulas ru from $.90 to $1.00 a ton for normal brightness an from $1.15 to $1.25 where premium brightness (u to 75) is required.

*CONTACT ONE OF THESE MEN FOR INFORMATION:*

**Midwest Area**
W. L. Liike, Chicago, Ill.—INdependence 3-7250

**New York & Boston Area**
C. R. Lombard, New York, N. Y.—LOngacre 3-6440

Or call: A. T. Hawkinson, Wilmi




"Solozone" sodium peroxide and "Albone" hydrogen peroxide.

# save up to $1.25 a ton over hydrogen peroxide alone

**ONE-STAGE TOWER PROCESS FOR PREMIUM BRIGHTNESS**

**A. USING COMBINATION OF PEROXIDES**

5.0% silicate
1.5% "Solozone" sodium peroxide
1.25% "Albone" 50 hydrogen peroxide

**B. USING HYDROGEN PEROXIDE ONLY**

5.0% silicate
1.5% NaOH
2.5% "Albone" 50 hydrogen peroxide

**ONE-STAGE TOWER PROCESS FOR NORMAL BRIGHTNESS**

**A. USING COMBINATION OF PEROXIDES**

5.0% silicate
1.2% "Solozone" sodium peroxide
0.7% "Albone" 50 hydrogen peroxide

**B. USING HYDROGEN PEROXIDE ONLY**

5.0% silicate
1.2% NaOH
1.7% "Albone" 50 hydrogen peroxide

Du Pont technical representatives will gladly show you how a combination of peroxides can save you money in your bleaching operation and suggest a process layout (such as the one shown above) to fit into your present plant layout. Contact your nearest representative listed below, or write: Du Pont, Electrochemicals Department, Wilmington 98, Delaware.

**West Coast Area**
P. E. Kiefer, Portland, Oregon—CApital 7-1281

**Southern Area**
R. W. Hammond, Charlotte, N. C.—FRanklin 5-5561

laware—PRospect 4-4698

**DU PONT PEROXIDES**

**ALBONE®** hydrogen peroxide

DU PONT REG. U.S. PAT. OFF

**SOLOZONE®** sodium peroxide

BETTER THINGS FOR BETTER LIVING ... *THROUGH CHEMISTRY*

# Paper and Pulp Mills Everywhere

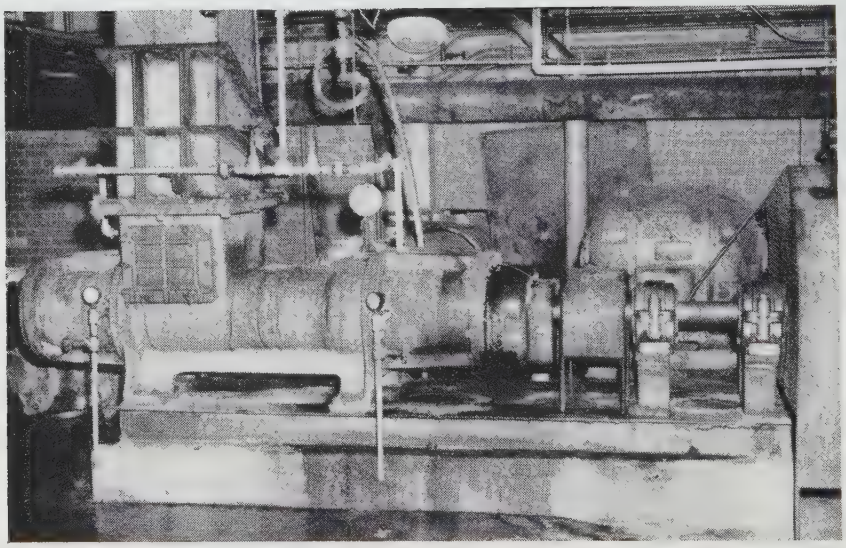
—IN MAINE, NEW YORK, PENNSYLVANIA, MONTANA, MINNESOTA, IDAHO, SOUTH CAROLINA, MARYLAND, VIRGINIA, GEORGIA, ALABAMA—

## are using Warren HIGH DENSITY SCREW PUMPS

## to move all types of high density stock cleanly, efficiently, most economically

Capacities to 750 tons per day; pressures to 300 PSIG; densities to at least 18%; no need for auxiliary feeding; no pulsation. These are a few of the production features of the Warren High Density Screw Pump that promise greater pumping economy...with any type of stock, in any locality.

*In the South,* one of the well-known installations that is producing to full planned capacity is at Union Bag-Camp Paper Corp., Savannah, Ga.



Warren No. 11 High Density Pump; up to 455 GPM; 12-15% washed hardwood pulp from washers to storage.

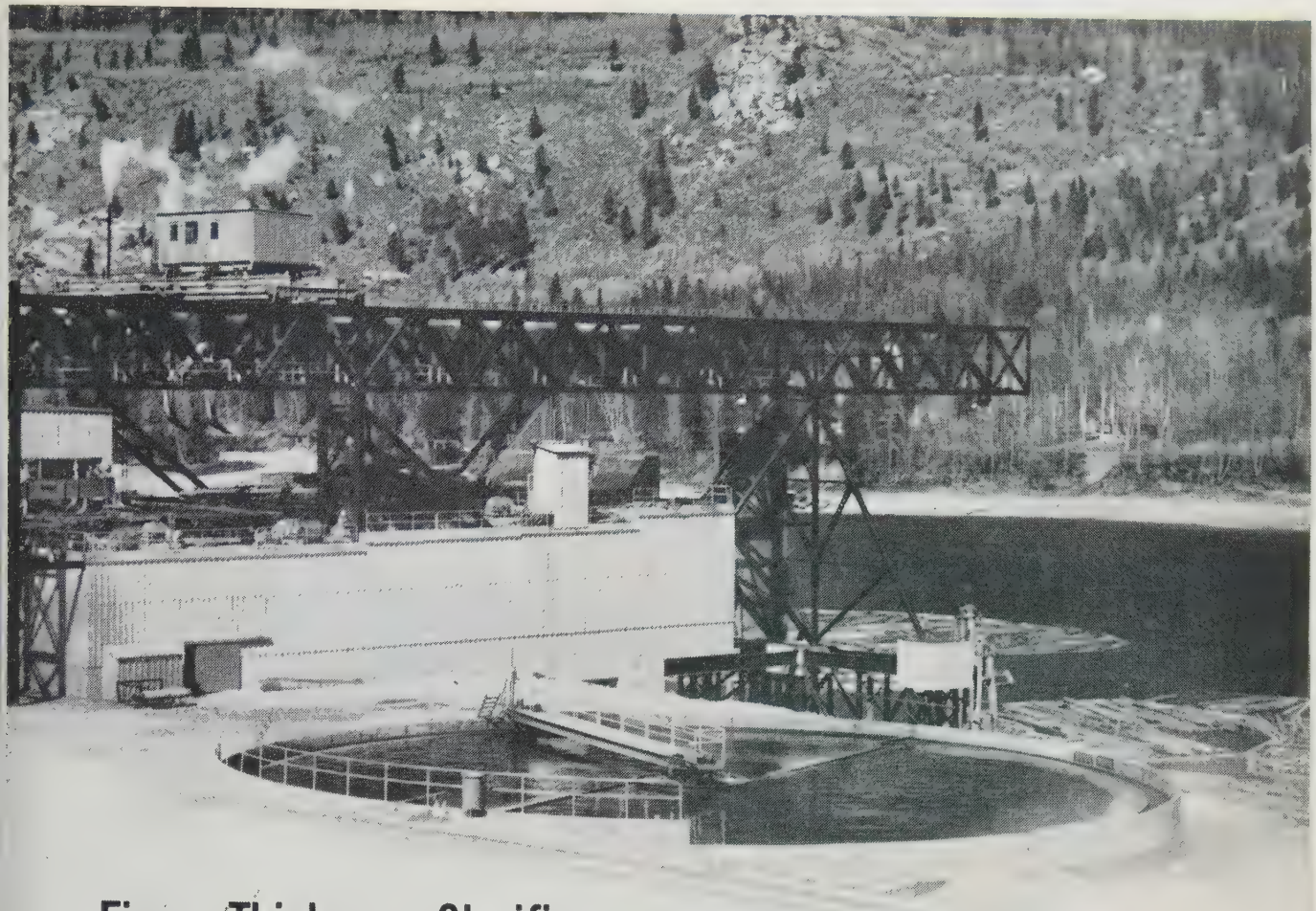
Ask for information, including a detailed listing, on how other mills throughout the country are using the versatile Warren High Density Screw Pump. And remember that Warren engineering is always ready to help with the most economical solution to your special pumping problems.

WARREN

PP-45

CENTRIFUGAL · SCREW · RECIPROCATING · GEAR

**WARREN PUMPS, INC.** WARREN, MASSACHUSETTS



# Eimco Thickener Clarifies Barker Water at New Celgar Kraft Mill

When Celgar, Limited, Castlegar, B.C., planned their new kraft mill, recently put into operation, they chose a modern 100-ft Eimco-Process Type C thickener for removing bark, sand, float material and chips from 4000 gpm hydraulic de-barker water.

The Eimco thickener is a steel center column supported unit with double skimmer. For convenience, feed enters at the bottom of the 24-ft center column. Underflow solids are dewatered on an Eimco 6-ft diameter by 7 disc filter; overflow goes to the Columbia River; float is burned.

In engineering this industrial type clarifying thickener for economical barker water clarification, Eimco engineers worked closely with engineers of H. A. Simons, Ltd., Vancouver, B.C., consulting engineers for the Celgar pulp mill.

The Eimco representative in your area can provide facts about Eimco equipment that has proved profitable for pulp and paper producers—equipment for wastes and water treatment . . . pulp washing . . . filtration . . . recausticizing. Contact him, and write for Thickener Bulletin SM-1004.

The Eimco Corporation, Box 300, Salt Lake City, Utah.

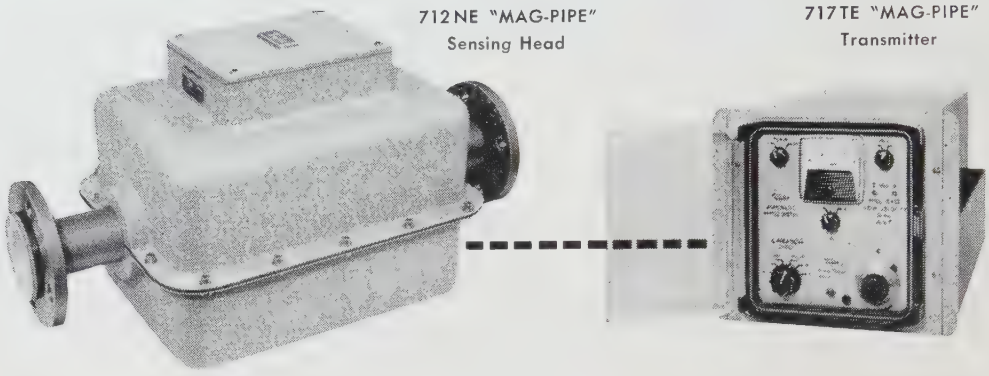
EIMCO

"Advanced Engineering and Quality Craftsmanship Since 1884"

B-843

# "MAG-PIPE"*

# MAGNETIC FLOW TRANSMITTER




## For Stock, Caustic, Size, Alum, Clay, Starch, Separan, Melamine

In addition to the "difficult-to-handle" fluids listed above, the MAG-PIPE Flowmeter is ideal for measuring flow of white water, black liquor, titanium oxide . . . practically any liquid with an electrical conductivity as low as that of distilled water. As it presents no restrictions to flow, very low flows, viscous liquids, and liquids containing solids are no problem.

The MAG-PIPE Transmitter sends signals up to 10,000 ft. with ½% accuracy. When used with miniature receiving instruments, such as TRANSCOPE® Recorder Controllers, it saves expensive panel space and makes centralized control rooms highly practicable. For versatility of application, the MAG-PIPE Flowmeter is designed in components which can easily be installed in an existing system.

Its sensing head mounts in the flow line. It comprises a flow pipe, an AC magnetic circuit and two electrodes. Sensing head housing is "hose-down proof" (explosion-proofing optional), and has lap joint (Van Stone type) flanges for easy installation.

The transmitter employs solid state components, being fully transistorized for long-term reliability. It is standard in fixed-range form, in which case calibration is factory set at the desired range. Optionally available with a full-scale vernier adjustment and a 2-position range switch to measure any range span between 0-½ and 0-30 ft./sec. A built-in output meter doubles as circuit checking device.

• • • •

The MAG-PIPE Flowmeter Transmitter is in production. Consult with us on your difficult flow measurement problems. See your Taylor Field Engineer or write for **Bulletin 98418.** Taylor Instrument Companies, Rochester, N.Y., and Toronto, Ontario.

### OUTSTANDING FEATURES

- **High accuracy—1/2% of full scale with most ranges**
- **Full scale velocities as low as 1/2 ft./sec. at only slightly reduced accuracy**
- **Output signal—1-5 ma DC into any load from 0-3000 ohms**

**(Electro-Pneumatic Transducer or TRANSCOPE Electronic Control System)**

*Trade Mark

*Taylor Instruments* **MEAN ACCURACY FIRST**

# Papermaker's Rx for the Blues

## NATIONAL®

# Brilliant Bond Blue NMP

***Another new development from National Aniline paper dye research!***

One more good reason why mills look to National Aniline for leadership in the technology of profitable colored papers!

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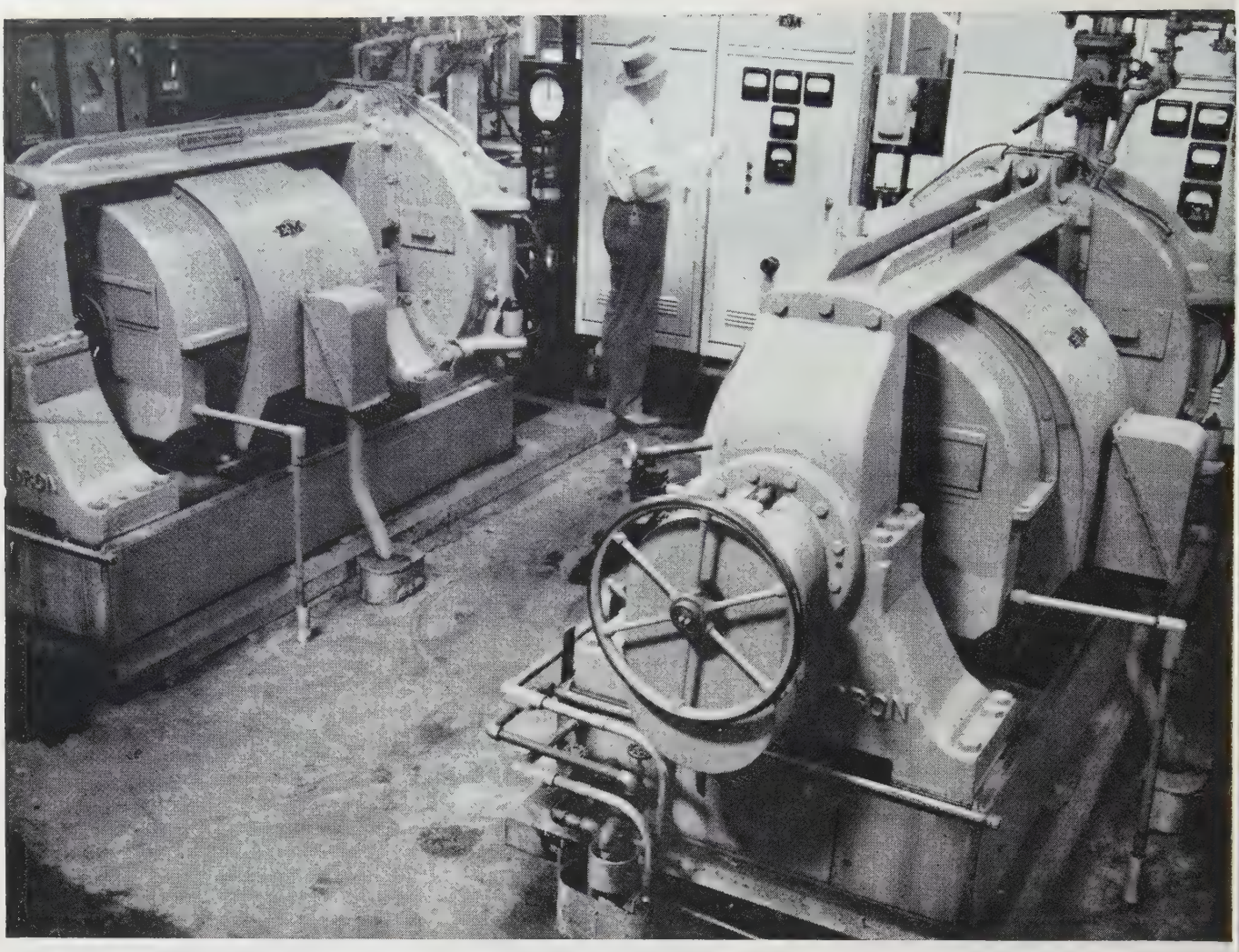

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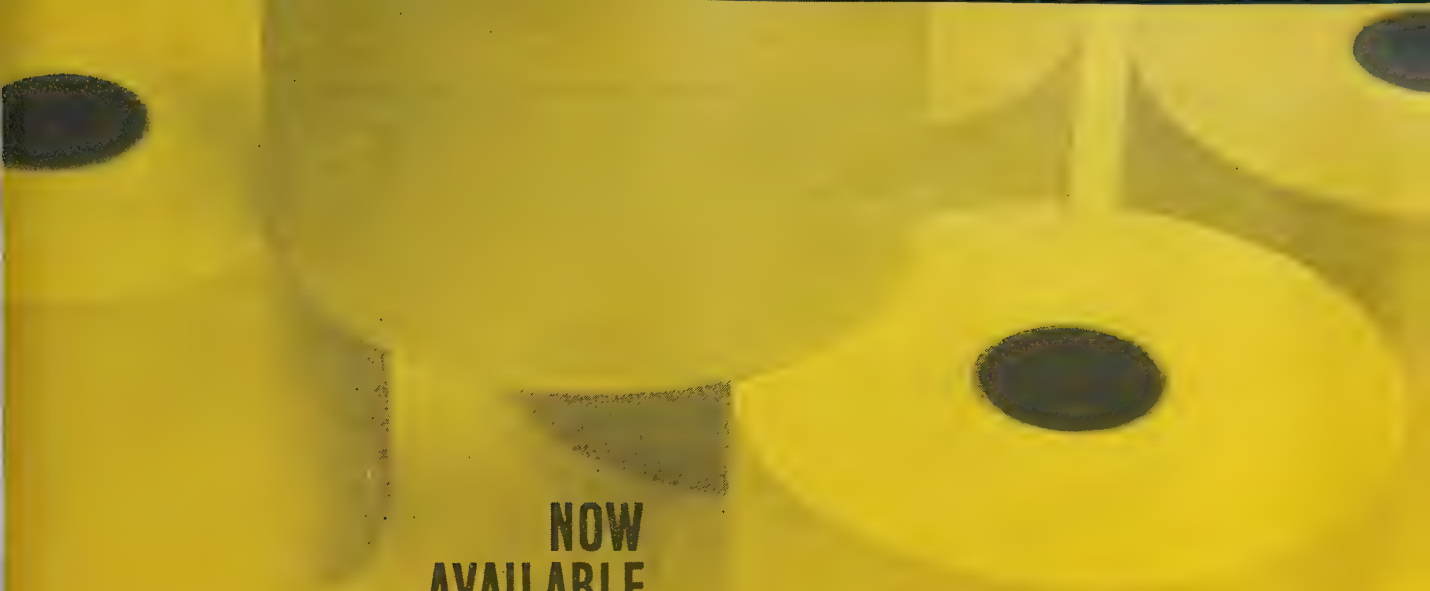

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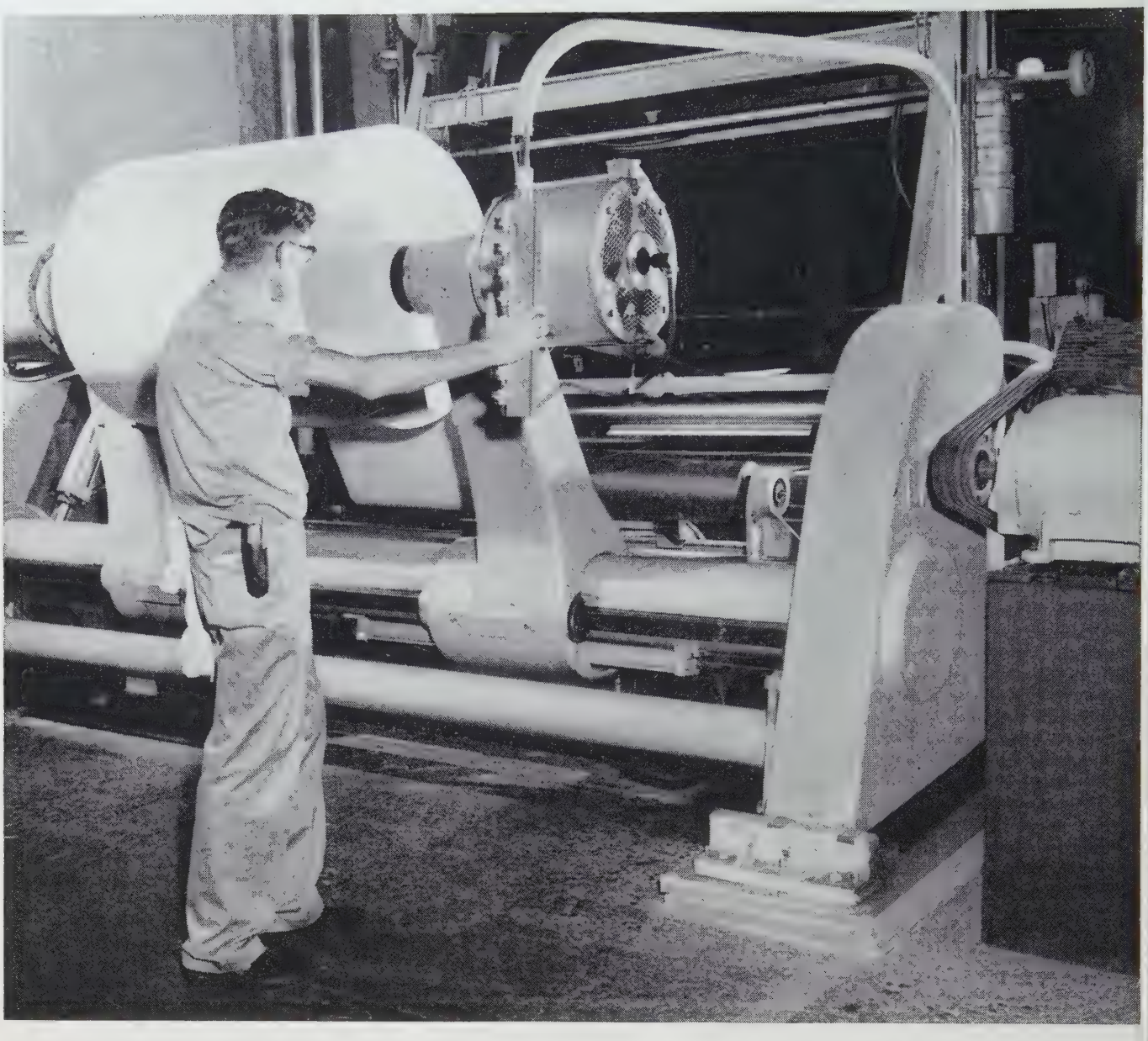

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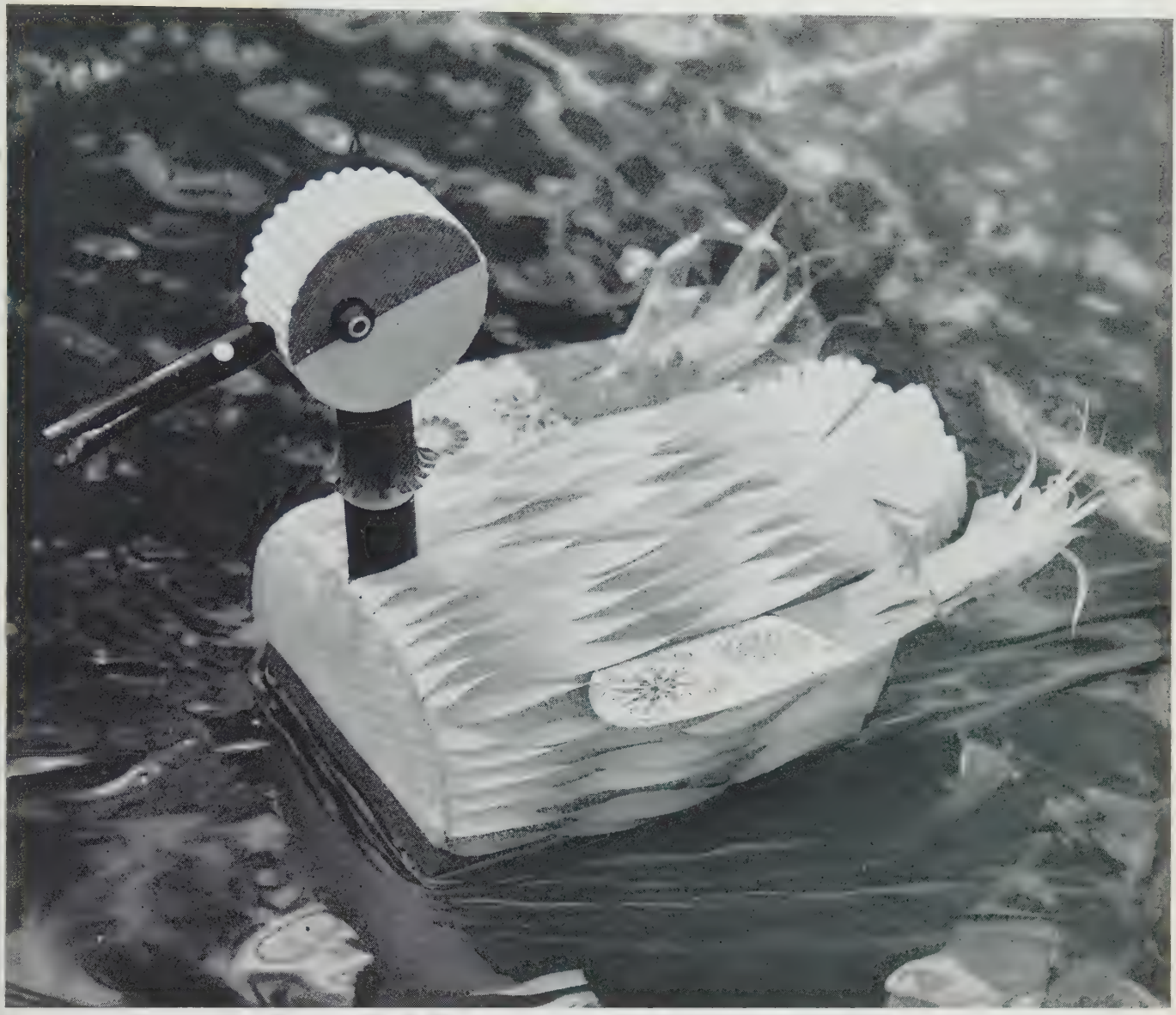

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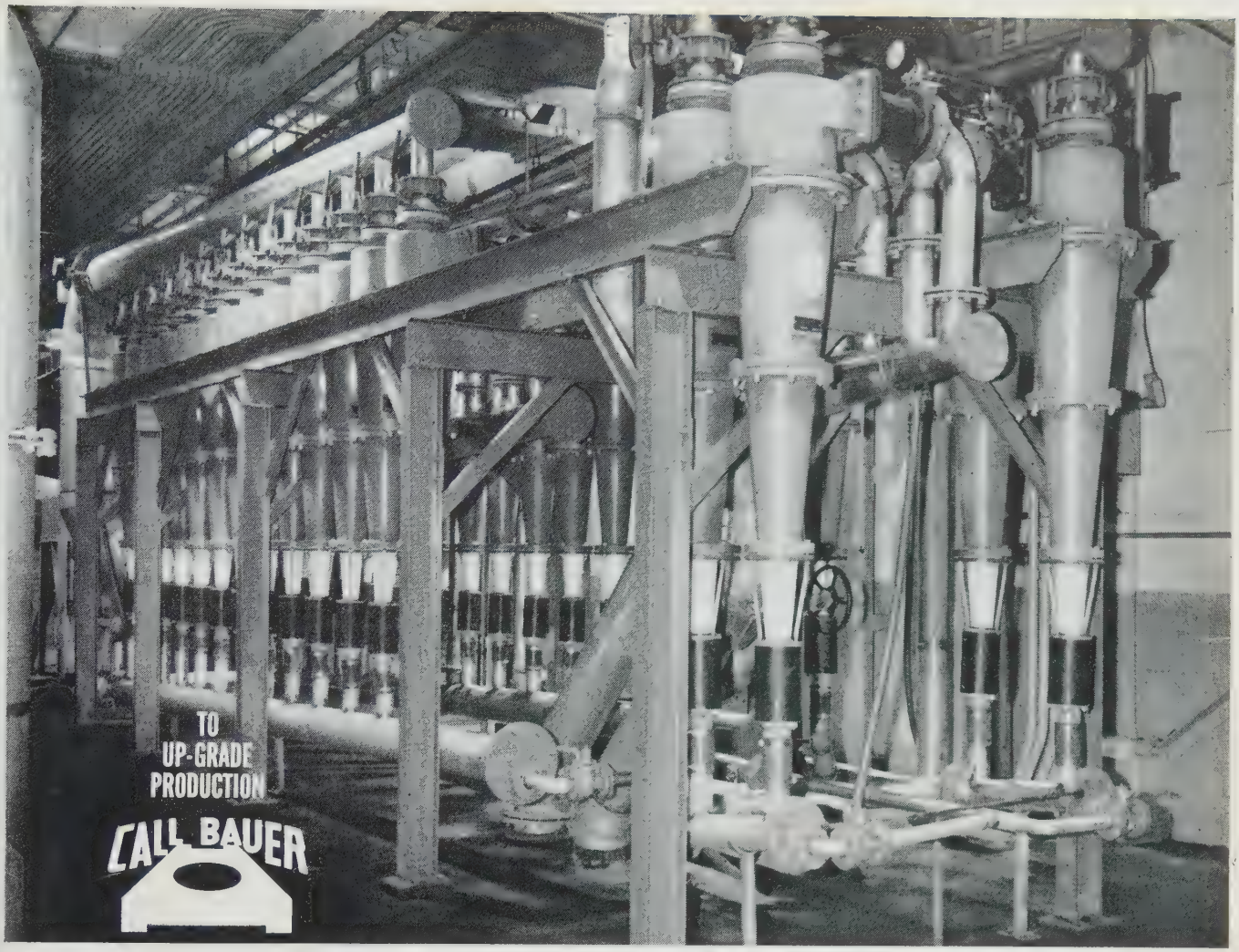


Battery of Bauer No. 623 Centri-Cleaners on No. 4 machine at St. Croix Paper Company. Note long lasting ceramic lower cone nozzles and vacuum rejects-separator system which reduces wear on cleaners in the second and third stages of this modern installation.

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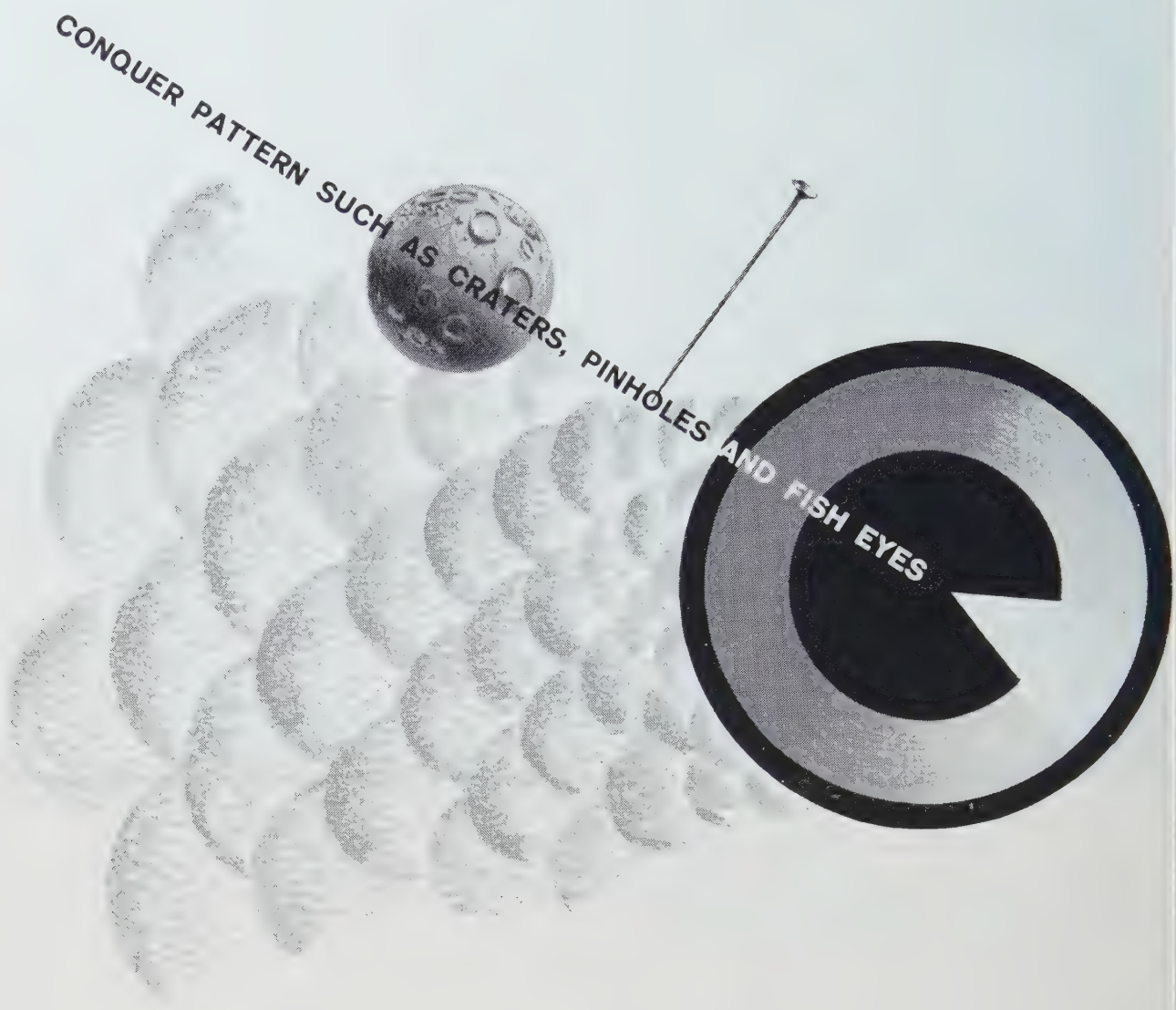


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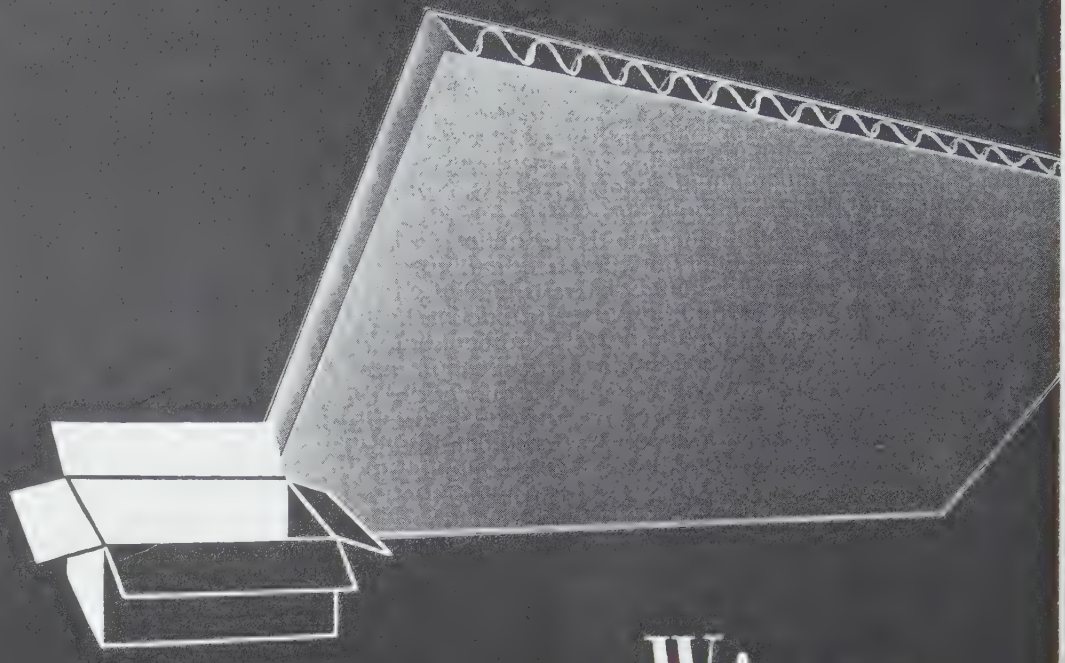

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# Instrumentation as Applied to Drier Drainage and Moisture Control Systems

H. P. FISHWICK

**Typical instrumentation associated with cascade type paper machine drainage systems and also those employing thermo-compressors as a means of promoting drier evacuation are discussed. With machine speeds approaching 3000 f.p.m., adequate control of all machine variables is a necessity in order to obtain condensate removal and quality control of the final product. The use of moisture control systems is an attempt to control the moisture content of the web as it leaves the machine and also ahead of the size and coating sections. It is one of the most difficult variables to measure because of the many other process variables that directly affect it. This paper attempts to explain the basic operating principles of some of the systems now commercially available for control of sheet moisture.**

REGARDLESS of the type of paper manufactured, the basic machine problems are the same; namely, to remove the condensate and noncondensable gasses from the driers under all operating conditions and to maintain reasonable steam economy on the machine while obtaining the flexibility of operation desired. Unfortunately, such results are not always compatible.

The use of correct instrumentation on the drying section of a paper machine is an attempt to minimize mechanical operating problems, to remove the human element, and to promote repetition of product quality. This is accomplished by controlling pressure, temperature, siphon differentials, liquid levels and vacuum, if such a system is used.

## CASCADE DRAINAGE SYSTEMS

The most prevalent type of drainage system being used today is commonly referred to as a "cascade" or "blowthrough" type system. This system cascades the blowthrough steam through succeeding drier sections to a small group of driers at the machine wet end. The last stage discharges to a condenser, or possibly the steam is used in some other mill process. The final use of the steam will depend to some extent on machine operating pressures and mill conditions. We should like to show two or three variations of the cascade type of system, and then discuss first, the instrumentation generally used for controlling steam admission to the various machine sections, and next, the means of controlling condensate evacuation.

The machine shown in Fig. 1 could be considered as employing the simplest of cascade systems. The only way it could be less complex would be to remove all the instruments and valves with the exception of the main steam controller. This has been done. Differentials were obtained by balancing the driers in each steaming section so that they would condense the steam from succeeding sections.

H. P. FISHWICK, Manager, Pulp and Paper Mill Dept., Mason-Neilan, Norwood, Mass.

A machine employing a system such as this would have to operate at a relatively constant speed and tonnage. Any change in machine speed would require a new differential pressure across the siphons for optimum operation. As you no doubt have surmised, a machine requiring a wide range of operating pressures could not operate in a very satisfactory manner if it were laid out in this fashion.

A somewhat more complex machine is shown in Fig. 2. The section of driers following the breaker stack is a simple two section cascade system controlled in exactly the same manner as the machine shown in Fig. 1, except with one less cascading section. The driers ahead of the breaker, sections two and three, are controlled in a manner similar to the dry end section.

There is one difference, however. The differential pressure across the section two driers is controlled by

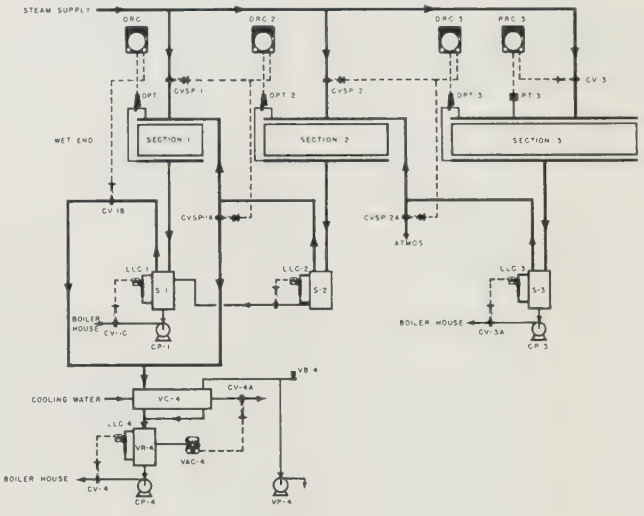


*Fig. 1*

means of two sequentially operated differential valves. One valve controls differential by discharging the blowthrough steam to the wet end header and the second valve discharges to the vacuum system. The number of driers in the wet end group is balanced with the section two driers so that there will always be a requirement for makeup steam through valve CVP-1. The steam pressure in the header supplying the six wet end driers can be set within 1 or 2 p.s.i. of the pressure in section two header and still not interfere with the evacuation of the section two driers. Valve CVSP-2A will throttle the section two blowthrough steam to the vacuum system, should it be in excess of the wet end condensing rate under certain operating conditions. The vacuum system on this machine is typical of most machine installations.

In the installation shown in Fig. 3, it was a requirement that the drier temperature gradient on the wet end be controlled by the operator. In addition, it was stipulated that normal temperature difference between two cascading sections could not be tolerated. To overcome this, three driers between the two cascading sections are controlled by differential control, with the independent variable being the main steam header pressure.

The slope of the gradient is changed automatically if a greater differential is needed between the two cascading sections. This is accomplished by means of remote loaded pneumatic set units in each of the differential instruments. The lead driers on the wet end could be controlled in the same manner if desired. There are many variations of the systems shown and also other methods of applying instruments to accomplish the same end result.

## DIFFERENTIAL PRESSURE CONTROL

The proper evacuation of the machine driers can be assured only by maintaining a positive pressure differential across the siphon. The differential must be great enough to remove all condensate as it accumulates. The differential will also remove noncondensable gases as they accumulate in the drier. If these noncondensables are not removed, steam temperature within the drier will be depressed. Continuous purging of steam will also promote circulation within the drier.

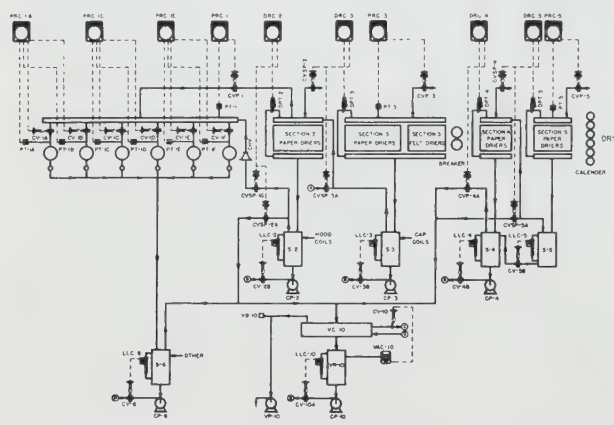

*Fig. 2*

The required differentials are a function of speed, drier diameter, and siphon design. The rotating type siphons usually require a higher differential than the stationary type because of the necessity to overcome the centrifugal force developed by the rotating siphon.

We do wish to point out the fact that the blowthrough steam from each drier of a cascade system is determined by the differential across the siphon. The required differential has been determined by trial and error at a given machine speed and the blowthrough steam is a function of this differential. We do not actually control the blowthrough to some predetermined value. Naturally, we can reduce differentials if they are too high and thereby reduce steam blowthrough. However, we cannot cut back beyond the point where drier evacuation is disrupted. Decreasing differentials too far can result only in water logged driers.

In the automatic control of differential pressure across the siphons, a differential transmitter and receiving controller are usually employed. The transmitter senses the difference in pressure and relays a 3 to 15 p.s.i. air signal to the receiver controller instrument. This signal is a function of the pressure differential. The output pressure of the differential control instrument strokes the control valves, which are sequentially operated makeup and blowdown valves.

In referring to the simplified machine in Fig. 1, the makeup valve, CVSP-2, which is usually air-to-open, would operate on a 9 to 15 p.s.i. portion of the total instrument output of 3 to 15 p.s.i.; whereas the blowdown valve, CVSP-2A, which is air-to-close, strokes on 3 to 9 p.s.i. of the total 3 to 15 p.s.i. output signal of the differential instrument.

With the machine operating under a load, the blowdown valve would be shut and the makeup valve would be in a throttling position. The blowdown valve functions only during startup and during machine breaks.

In new paper machine installations, the use of differential instruments and control valves is generally considered to be superior to the use of fixed orifices and traps on the driers, particularly on machines designed to operate over a wide range of pressures. A machine that employs traps, or any nonautomatic restricting device, is not ridding itself of noncondensable gases in a manner that would promote the best rate of heat transfer across the shell, or even temperature across the drier face.

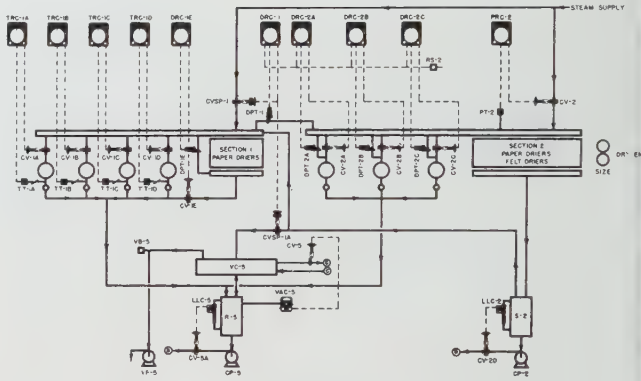

*Fig. 3*

In instances where restrictive devices of all kinds were removed from the drier condensate lines, improved operation was obtained in the form of higher surface temperature and minimizing of cross-machine moisture variations. We must point out, however, that in most cases an increase of steam consumption was the result. It is not always possible to obtain low steam consumption with machine flexibility and still end up with a quality product at the reel.

On most steaming sections, some type of separator is used for removing the condensate from the blowthrough steam so that relatively dry steam passes to the wet end driers. On some sections, it is expedient to control the differential by throttling a control valve installed in the condensate and blowthrough steam line. When this is done, the use of a chromium-molybdenum or stainless steel valve body is recommended. Unfortunately, good, clean water has an affinity for cast iron or steel; and erosion can take place with pressure drops as low as 30 p.s.i. under certain conditions.

## VACUUM SYSTEM

In further considering paper machine drainage systems, we feel that too little thought is given to the designing of an adequately sized vacuum system. On machines operating at low pressures, the need for adequately sized condensers cannot be overstressed. Many mills require independent operation of the first few wet end driers in order to control the temperature gradient. The use of graduated low surface temperature in this region can be of great assistance in overcoming picking and cockling problems; and, without a properly sized vacuum system, low drier temperatures are impossible.

The use of a condenser on a paper machine is similar to its use on a turbine. The prime reason the condenser is used on a turbine is to reduce the back pressure so that maximum heat energy can be extracted from the steam. Thus more power is obtained. When used on a paper machine, the condenser reduces the back pressure on the machine, thereby permitting condensate removal at much lower operating pressures.

A condenser must be capable of removing sufficient heat from the blowthrough steam to create the minimum required vacuum. The vacuum pump on such a system must be capable of removing the noncondensables at the vacuum created, discharging these noncondensables to atmosphere. The two methods that have been used to condense the blowthrough steam are the direct contact type and the surface type condenser.

The direct contact type utilizes a spray nozzle to mix cooling water with the steam. This method is undesirable for two reasons; first, treated cooling water is required, and second, a spray nozzle cannot be automatically controlled and still retain its spray pattern.

The only reasonable way to design a spray system, assuming the cooling water can be introduced into the condensate, is to design for maximum operation at all times. However, the most flexible system employs the shell and tube type condenser, where cooling water can be throttled to maintain the desired system vacuum.

The vacuum pump must be sized to remove all the air and noncondensables as they accumulate in the condenser. If this is not done, the efficiency of the condenser is greatly reduced. In fact, 5% air entrainment by weight will reduce the heat transfer rate of the condenser by about 50%.

## VALVE AND LINE SIZING

In the overall design considerations of a paper machine, the sizing of valves on the steaming side of the machine is relatively simple. Steam admission valves are sized to meet the drying demand, while the valve plug is at a reasonable throttling position.

On the condensate side of the machine, sizing of lines and supplementary equipment becomes somewhat more critical.

Separator tanks and lines must be sized for minimum operating conditions due to the large volume of steam being handled at the low operating pressures. If this is not done, severe losses will occur that will affect drier differentials when operating at minimum conditions. Steam separator tanks not only act as reservoirs for the condensate, but also are designed to reduce the steam velocity for separation of steam and condensate.

Control of level in the separator is usually accomplished by means of a displacement type level controller which throttles a condensate valve in the pump discharge line. This, of course, holds the required minimum NPSH on the pump suction.

## JET COMPRESSORS

We have discussed the relatively simple three section cascade system and some of the variations of this basic system. Also, we have shown some of the standard methods of instrumentating paper machine drying sections. There are many variations of what we have shown.

Now we turn to the jet compressor as a means of promoting circulation in a paper machine.

Figure 1 shows a three section cascade system. We can easily see that with high speeds, requiring high differentials, considerable difficulty would be encountered in the evacuation of condensate if the dry end section could only operate at 15 to 20 p.s.i. It would be impossible to evacuate condensate from driers because a vacuum system could not be designed to reduce the back pressure on the machine to a sufficiently low level. The use of a jet compressor helps to overcome this condition.

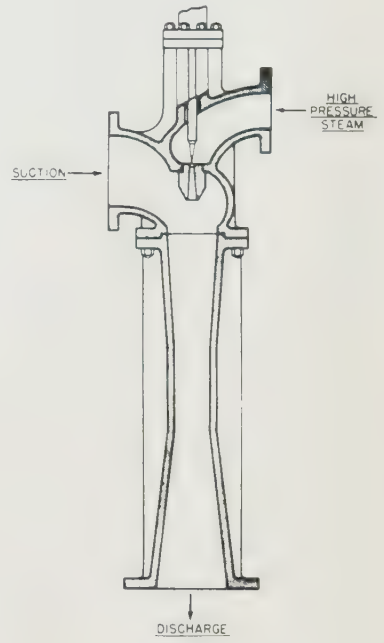

*Fig. 4*

Jet compressors were first used on Yankee drier machines to recirculate the large quantities of blowthrough steam. This was an economy measure because blowthrough rates of 7000 to 8000 pph were not uncommon and it does become somewhat difficult to utilize these large quantities of blowthrough steam in other mill processes. Recirculating the steam was the answer; hence, the use of the jet compressor.

The actual operating principle of the jet compressor is quite simple. The actuating medium, which in our case is high pressure steam, enters the compressor and flows through a nozzle at a velocity in the order of 3000 to 5000 f.p.s. The high velocity steam entrains the low pressure steam in the suction chamber and then passes through a diffuser where the velocity energy is converted to pressure energy. The exit pressure is consequently higher than the pressure at the jet suction.

Two types of compressors are currently in use: the compressor with the fixed nozzle where the motive steam is throttled by means of a control valve in the steam supply line, and the compressor that utilizes a movable spindle at the nozzle. Both types of units are being used successfully. Figure 4 illustrates the jet compressor utilizing the movable spindle. The advantage

of using these units on a paper machine is that each section of driers can be independently controlled.

This means that our three section cascade system, previously described, can now consist of three or four independently controlled sections, all running at the same pressure if necessary.

We believe the greatest benefits from this type of system are derived on a newsprint machine operating at high speed, with rotating type siphons and low operating pressure. There is no question, however, that they can be used to advantage on machines manufacturing other grades of paper.

The initial use of compressors employed a system similar to that which had been used successfully on Yankee type driers for many years. Figure 5 shows the instrumentation as applied to one drying section only. Other variations of this have been successfully used in recent years. One system that has now been in operation for almost two years is shown in Fig. 6.

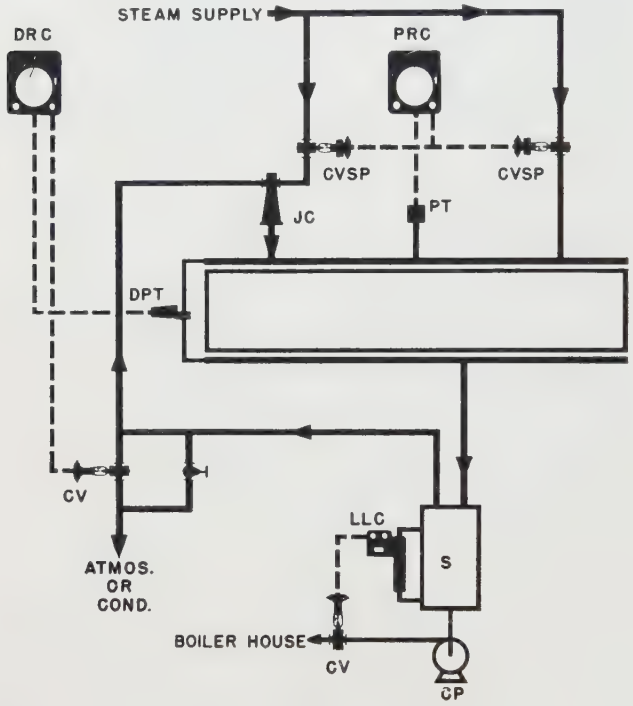


*Fig. 5*

This shows one drying section of a machine that is controlled in the standard fashion as far as steam admission is concerned. However, a differential instrument is throttling the high pressure motive steam to the jet compressor in order to hold a fixed differential at all times.

We control this single dry end section in this manner for two reasons: first, to obtain all the drying capacity possible from this section of driers, and second, to keep the usage of 150 p.s.i. motive steam to a mimimum. By controlling the section in the manner shown, the regular mill source of 60 p.s.i. steam is used as makeup, and only a minimum quantity of high pressure steam is needed to recirculate the blowthrough steam. Maximum drying pressure in the section is 50 p.s.i.

Many variations of the jet compressor system are possible, and the use of multiple compressors on one section of driers to obtain a wide operating range is being utilized on several machines.

## MOISTURE SYSTEMS

On all the diagrammatic drawings shown, steam admission to the main drying sections has been shown as pressure control. This is probably the most prevalent type of control being used today.

The pressure controller is set to maintain a fixed pressure in the steam header by throttling a steam admission valve. The instrument will maintain this pressure until the set point is changed.

A method of automatically changing the instrument set point in order to maintain a relatively constant moisture in the web is by means of a device that detects moisture changes in the sheet.

The measurement of moisture changes in a sheet of paper running at speeds of 2000 to 2500 f.p.m. is most difficult because many other variables in the paper-

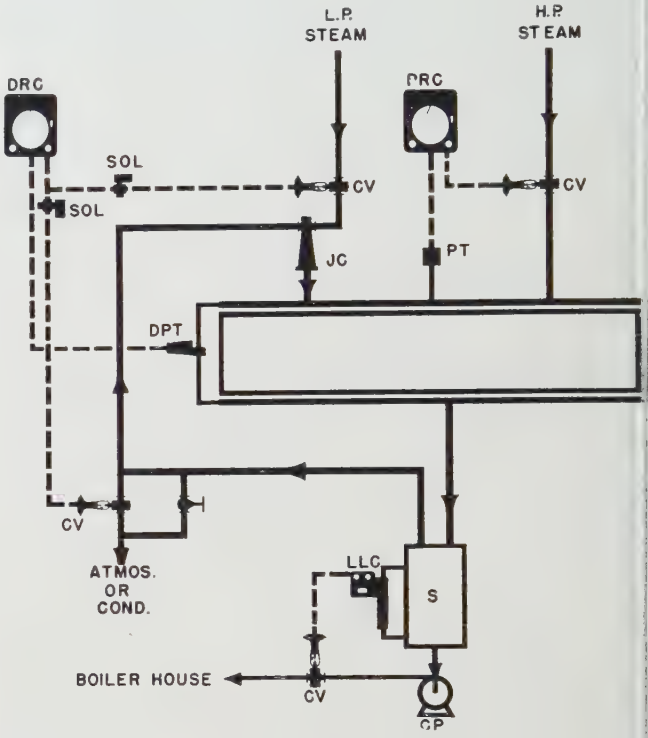


*Fig. 6*

making process affect the final moisture of the sheet. The common tendency in most paper mills is to overdry the sheet, which can result in a brittle fiber of decreased strength and possible sheet discoloration. The use of a moisture control system is an attempt to overcome these problems and to remove some of the human element from this process.

We shall attempt to present the operating principles of several commercially available moisture control systems. It is not the intent to cover all makes, but only a representative few. A detailed description of the various moisture control systems may be found in TAPPI Project 775 as presented by Robert Hurm at the 15th Engineering Conference in Jacksonville, Fla. This was a project initiated by the process instrumentation committee.

## TENSION ROLL TYPE

We believe the oldest method of controlling moisture is that employing a tension roll. This type of control

has passed through many stages of refinement from the original operation where the tension roll actuated the steam admission valve by means of pulleys and levers. Later, when pneumatic instruments and valves came into use, the tension roll operated a pneumatic pilot which in turn actuated the steam admission valve This type of control operates on the expansion and contraction of the sheet, caused by sheet moisture variations.

It has the advantage of averaging out the sheet moisture because the tension roll extends across the sheet width. Also it is unaffected by machine speed, sheet formation, weight, and ambient temperature.

The above description applies to the current Stickles Press-Ten-Trol system. Many systems employing the tension roll type of control have been used over the years. Both Pickle and Mason-Neilan once marketed such a system.

### ELECTRICAL CAPACITANCE TYPE

Another type of moisture control system as manufactured by the Foxboro Co. uses the principle of electrical capacitance measurement.

This system employs a three foot measuring head riding on the top of the sheet so that it will not interfere with machine operation, either during the threading operation or during a paper break.

The measuring head is an electrical capacitor which uses the paper web as part of the measuring circuit. Electrical capacitance is directly proportional to the dielectric constant of the material. Therefore, changes in moisture of the sheet as it passes through the field changes the dielectric constant. The moisture control instrument resets the set point of a pressure or a temperature controller which in turn, throttles the steam admission valve.

The system also employs an automatic break control which reduces the drier temperature. It then automatically returns the set point of the pressure or temperature controller to its required value, when the sheet is returned to the machine. As a standard, this system will work successfully on sheet moistures up to approximately 15%.

### TEMPERATURE MEASUREMENT TYPE

The Hurletron system operates on the principle of sheet temperature measurement. The moisture measurement takes place at the end of a drier section. Multiple gages are spaced across the sheet so that recordings of moisture variations may be obtained at several points.

One of the last driers of the section to be controlled is divorced from the steam system of that section and receives its own independently controlled steam supply. The reference drier is also trapped.

When the sheet leaves the reference drier, the moisture in the sheet vaporizes quite rapidly for a distance of 8 to 10 in. This vaporizing lowers the sheet temperature very rapidly. At a distance of about 30 in. from the reference drier, a sensing element is located. An element also contacts the surface of the reference drier.

A variation in the sheet moisture as it passes over the reference drier affects the temperature difference between the two measuring heads. This affects the output signal of the thermocouples in the measuring heads. One of the gages is connected to a pneumatic controller which throttles a steam admission valve to the drier section being controlled.

It is claimed that measurement is not affected by speed, size, pH, or sheet tension.

### PILOT DRIER TYPE

The two systems employing the pilot drier type of control are represented by both Stamm and Mason-Neilan.

These systems depend on the condensing rate change in the pilot drier to obtain a signal for corrective action to be taken.

The pilot drier is usually located toward the dry end of the machine section to be controlled. It can be ahead of the reel, size press, or coater. In some instances, it is located ahead of the breaker stack if that particular group of driers is to be controlled. It must be located at a position in the machine where there is a change in drier condensing rate. If it is located in a position where the sheet moisture is in the order of 2 to 3%, it is very likely that there will be no signal because the sheet surface temperature and drier temperature are very close. Under such conditions, the condensing load on the drier is very light or almost nonexistent.

The change in condensing load can be detected by means of a flow transmitter, or the change in pilot drier pressure can be detected by means of a pressure transmitter located at the pilot drier. Both methods have been successfully used.

The air output signal from the transmitter goes to an intermediate moisture control instrument and then to a pneumatic set bellows in the main pressure or temperature controller. In order to maintain a constant sheet moisture, the signal change in the bellows resets the set point of the main control instrument, thereby increasing or decreasing the flow to the machine.

### SUMMARY

Constant differentials must be maintained across the siphons at all times.

Maximum drier surface temperature and machine response to changing drying demands are the benefits obtained from a restriction-free machine.

Jet compressors are used on many machines to recirculate blowthrough steam which might otherwise be wasted. There are some operating limitations to the jet compressor, such as its rangeability and the fact that it forms a closed system with the machine section. This complicates the removal of noncondensables; their presence causing depressed temperature in the driers.

A moisture control system is used to hold the final sheet moisture within certain acceptable limits. This prevents possible sheet discoloration, and a brittle fiber, resulting in a sheet of decreased strength.

The correct selection of a moisture system will depend somewhat on the type of paper being manufactured and the proposed location of the system in the drier section.

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# Rheological Properties and Flow Modifiers for Trailing Blade Coatings

J. F. HERN

**Pigmented paper coating formulations applied by trailing blade coaters may contain one or more additives which improve the rheological properties of the formulation and impart additional surface effects to the coated paper. These additives may be metallic soaps, soluble soaps, waxes, fatty esters and ethers, sulfated fats and oils, fatty amines and amides. The effects of these products, along with other variables such as coating solids, formula types, base stocks, machine speeds, blade pressures and blade angles on flow properties, coater performance, and the finished coating have been studied. A Hercules high shear viscometer was used to examine flow properties and predict coating behavior, and a Time, Inc. bench model trailing blade coater was modified to permit correlation between laboratory and pilot coater studies. Results indicate that such correlations are possible and that the selection of an additive depends upon the desired flow and other coating properties.**

THROUGH the years of coating technology, additives have found extensive usage in low, medium, and high solids pigmented coating formulations to provide a wide variety of functions. The various additives or adjuncts which are employed belong to such chemical classes as soluble and insoluble soaps, waxes, fatty esters, sulfated oils and fats, organic esters, amines, and amides, which can be nonionic, anionic, or cationic in nature. Used only in relatively small amounts, they perform such feats as defoaming and prevention of entrapped air, leveling, and lubrication of the color itself, and also improve gloss, smoothness, elimination of pattern and spots, antidusting, fold, wet rub resistance, holdout, and improved printing properties, etc., to the coated sheet. Rheologically they function to allow adjustments in flow characteristics and per cent solids to suit the type of coating equipment, drying capacities, and formulation constituents.

For example, certain types of soluble soaps (sodium or ammonium stearate) and metallic soaps (calcium stearate) fatty esters, and gums are commonly used to increase thixotropy on print-type roll coaters, aiding in film splitting by allowing the coating to become thinner during the shearing action of each roll and giving the coating enough viscosity to remain on the rolls as it is worked nip to nip. In some low-to-medium solids applications, such as air knife coatings certain urea compounds, alkyl aryl sulfonates, and other fluidifiers can be used to lower viscosities and obtain near-Newtonian coatings at higher solids, thus permitting faster running speeds with limited drying equipment.

With the great trend to trailing blade coaters, and with many of the initial equipment and operative difficulties ironed out to a certain extent, makers of coated papers are looking toward additives and improved formulas to give them better coatings. In trailing blade coating operations, we are dealing with high solids coatings ranging from low through extremely high viscosities, depending upon the application involved.

This brings up the question: "What type of additive improves the rheological properties of trailing blade coatings?" Or, "What type of additive used to improve certain surface functions in trailing blade coatings has the least or greatest effect on rheological properties?"

These are the types of questions that we as additive suppliers asked ourselves. We decided that in order to study the effects of additives rheologically, we first had to study the effect of other variables, such as coating solids, machine speeds, blade pressures, blade angles, base stocks, and formula types on the flow properties and coat weights obtained. While there was very little printed matter available for background information, we found the work done by Follette and Fowells (*1*) on operating variables of blade coaters particularly helpful in orienting us for our studies. This paper gave us an excellent picture of what to expect from coater variables, however, it was rather limited in rheological studies, especially since high shear instruments were not used.

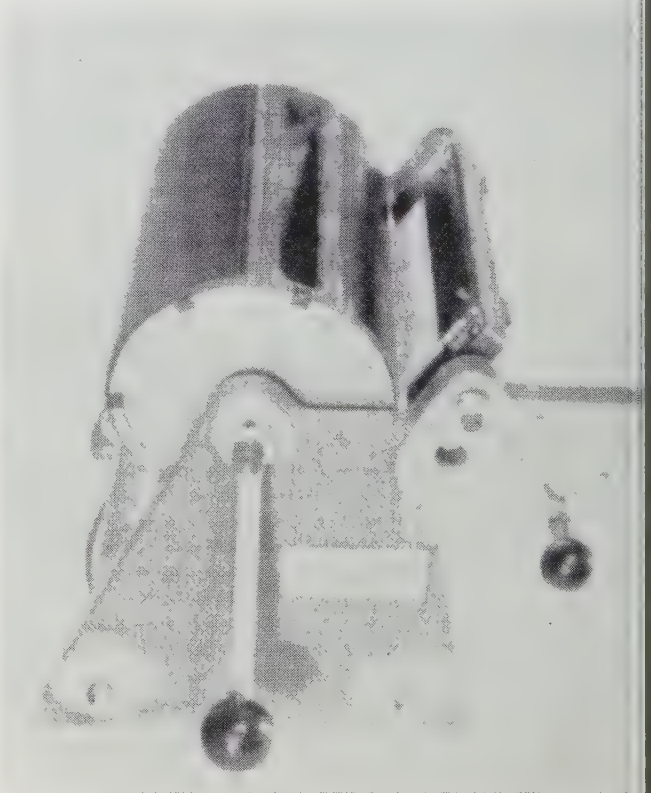

*Fig. 1. Bench model, hand operated, laboratory trailing blade coater*

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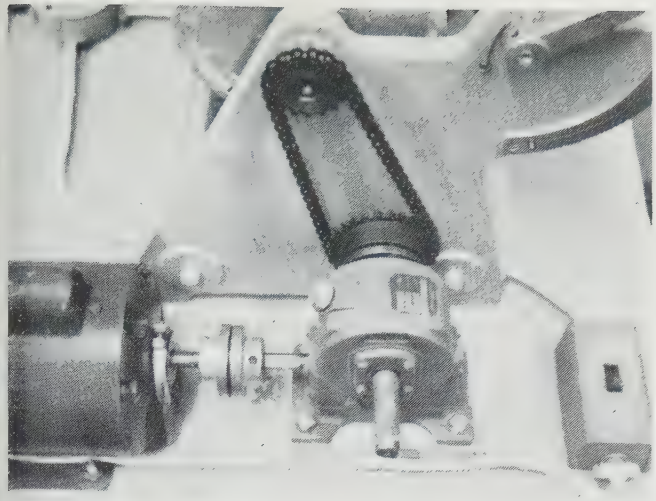

*Fig. 2. Modified laboratory trailing blade coater—electric motor, speed reducer, sprocket, and chain drive*

## LABORATORY STUDIES

### *Equipment*

*The Hercules High-Shear* rotational type viscometer (*2*) was chosen to enable us to study flow properties at higher shear rates. While this instrument does not approach the shear rates achieved by the action of the blade in the coater itself, it does give an indication of what to expect with non-Newtonian fluids and has the added advantage of producing plotted rheograms for study.

The next step was to find a laboratory coater which would correlate well with actual plant equipment. We felt that a good laboratory coater was essential since we wanted side-by-side comparisons of rheograms and coated papers with the same batch of coating and we had such a wide range of variables to study.

*The Time-Life bench model trailing blade coater,* manufactured by Precision Welding and Fabricating Co., appeared to offer many advantages, in that it was easy to use and gave quick results.

This piece of equipment (Fig. 1) as received from the manufacturer is a "puddle type" trailing blade coater and consists of a $10^{3}/_{4}$-in. long, 7-in. diameter rubber-covered backup cylinder fitted with an 8-in. long by $2^{1}/_{2}$-in. wide flexible blade of 0.012-in. blue spring steel

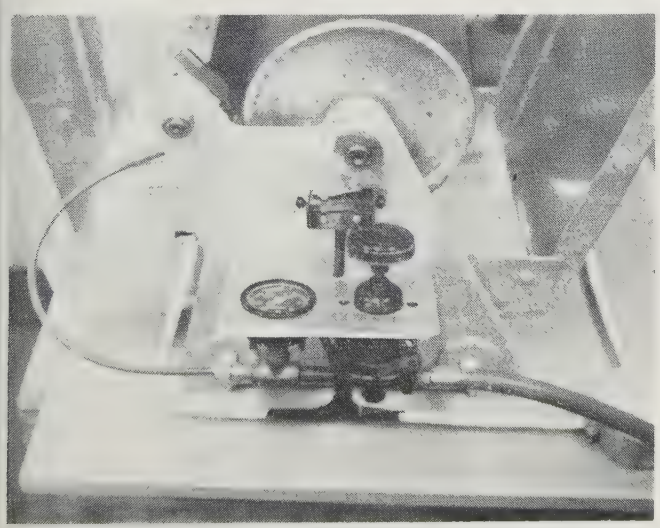

*Fig. 3. Modified laboratory trailing blade coater—cam detail, microswitch, pressure gage, and pressure control valve*

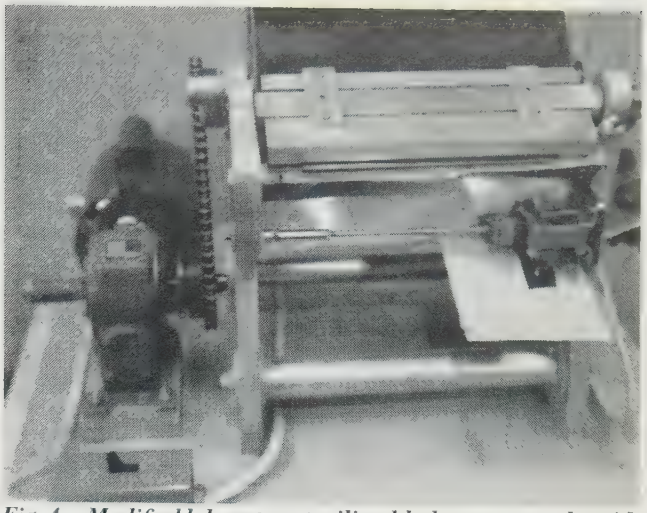

*Fig. 4. Modified laboratory trailing blade coater—solenoid, latch, and spring assembly*

supported by a stainless steel arm. The coating blade is held evenly against the backup cylinder by a rubber tube. The air pressure in the tube can be varied from 0 to 30 p.s.i.g. by a pressure-reducing valve and gage attached to a panel on the back of the coater. Thus, air pressure is used to vary blade pressure and coat weights. Sheets of paper or board (7 by 21 in.) are coated by attaching them to the backup cylinder with masking tape. The coating is applied to the leading edge of the sheet and spread evenly over the sheet by the coating blade as the cylinder is rotated past the blade. However, this lab coater is hand-operated by means of a crank, and therefore subject to wide variations in speed due to operator error. It was found that the Georgia Kaolin research department had taken some of the human element out of this coater by modifying it, utilizing an electric motor and speed reducer. This modified coater, however, could not be operated at higher speeds (in excess of 200 f.p.m.), because the blade had to be manually released in order to prevent double coating. It was decided that this problem could be solved by using an automatic blade release mechanism that would allow us to reach the speeds under which hydrodynamic pressures influence coat weight.

The Time-Life bench model coater has been modified by the incorporation of an electric motor, gear box, and chain device which turns the drum at approximately 500 f.p.m. (Fig. 2).

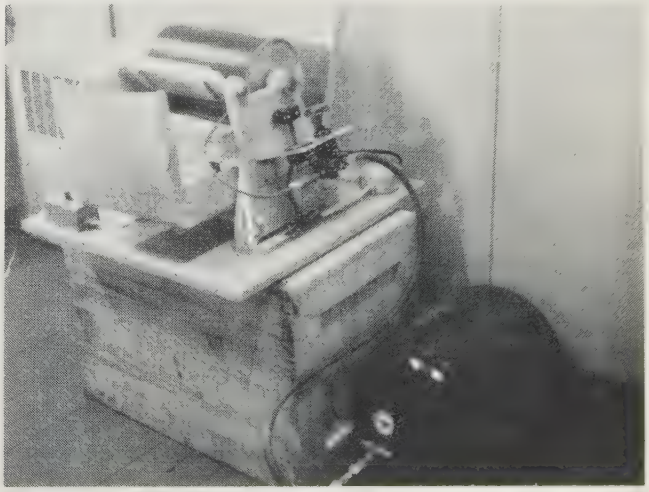

*Fig. 5. Modified laboratory trailing blade coater—overall view with splash pan and air supply attached*

In order to get the drum up to operating speed with the shortest possible acceleration period; a capacitor type 1/4 hp. motor with a high starting power was chosen. An Eberhardt-Denver 10 to 1 reduction gear box allows the motor to make several revolutions in accomplishing one full turn of the drum. This coupled with a sprocket and chain drive to eliminate slippage on startup limits the acceleration to the first 6 to 7 in. of the coated sheet. The coating on the last two-thirds of the sheet is therefore evenly distributed at the set speed of the motor and gear box.

The blade release mechanism (Fig. 3) consists of a cam on the drum shaft where the hand crank was removed. This cam activates a microswitch which energizes a solenoid unit. The core of the solenoid is connected by a rod to a latch assembly which releases the spring-loaded blade arm when the coating is about 2 in. from the end of the sheet. The latch and spring assembly shown (Fig. 4) have been substituted for the manually operated latch assembly which comes with the coater. A splash pan fabricated of aluminum stock has been added to catch the spray as the blade is released (Fig. 5).

Although the speed (approx. 500 f.p.m.) at which our laboratory coating was done, is perhaps the lower limit for most production equipment, it is probably not far from typical pilot plant capabilities and is well within the range where hydrodynamic pressure has an effect in "lifting" the blade. It is believed that higher speeds can be obtained; however, this as yet has not been fully investigated.

### *Coating Variables*

*Formulations.* Because of the wide range of pigments and adhesives offered to the industry, it was impossible to test them all. We, therefore, decided that a typical letterpress and a typical offset formula would be used that would be representative of trailing blade coatings throughout the industry. These formulas were as shown in Table I.

Although the adhesive-to-pigment ratios used may appear to some coaters as higher or lower than those of their operations, we tried to compromise as much as possible. Actually, we were able to show the same general trends (which will be discussed later) using higher and lower adhesive-to-pigment ratios. The calcium carbonate in the starch-latex formula was added to increase the brightness of the coating since our body stock was fairly low in brightness.

**Table I**
Letterpress Type (Starch-Latex)

| *Parts by weight* | |
|---|---|
| 75.0 | Predispersed clay (coating grade) |
| 25.0 | Calcium carbonate (coating grade) |
| 0.2 | Tetrasodium pyrophosphate |
| 22.0 | Converted starch (chlorinated) |
| 5.0 | Butadiene-styrene latex |
| | Water to 60–65% solids |
| | Offset Type[a] (Protein-Latex) |
| 100.0 | Predispersed clay |
| 0.1 | Tetrasodium pyrophosphate |
| 13.0 | Delta protein (low viscosity) |
| 5.0 | Butadiene-styrene latex |
| 2.6 | Dicyandiamide (optional) |
| | Water to 60–65% solids |

[a] Although most coatings for offset printing contain some type of protein insolubilizer, we did not include one in our formulation since each wet-rub agent has a different effect upon flow properties and would require separate study.

### *Coating Preparation and Application*

All laboratory coatings were prepared in a Reed sigma blade mixer, removed and mixed with a higher speed mixer to render them uniform and split into several smaller batches. One per cent (dry) on the dry pigment of each additive then was stirred in, the solids adjusted accordingly, and each mix allowed to stand 1 hr. in a water bath at 85°F., so that a uniform temperature was maintained. The standing period also minimized any entrapped air that might change the rheograms. Rheograms and coating weight–blade pressure readings were then run on each coating mix. A control was also included for each master batch of coating and run with the other mixes.

To allow ease of application and to prevent "skinning" of the coating during the conditioning period, plastic dispensers were used and the coating squeezed out onto the sheet for each run. Since 5 p.s.i. of air pressure were required to fill up the tube of the lab coater and "zero in" the blade, so that it was just touching the back up cylinder, blade pressure settings as recorded are actually differential air pressure between p.s.i.g. and the "zero in" pressure. Over 300 coatings were tested using both formulas over a wide range of blade pressures.

## RHEOLOGICAL CALCULATIONS

### *Starch Formulations*

The starch-latex formulations used exhibited the typical thixotropic-plastic flows which are customary with these types of colors. Green (*3*), along with the later work of Smith, Trelfa, and Ware (*4*), (*5*), (*6*), have given us numerical formulas for calculating plastic viscosities, yield stresses, thixotropic indices, and leveling indices in these systems.

After experimenting with all the bobs of the Hercules high-shear viscometer, we standardized on the *b* bob as giving the most reproducible and accurate rheograms.

In the example listed below, the constants for the *b* bob are $S = 0.0003262$ and $C = 0.01465$. The constant used for all rotational viscometers is 9.55.

$T_{1000}$ = Torque from upcurve at 1000 r.p.m. = $8.9 \times 10^5$ dyne-cm.
$T_{500}$ = Torque from upcurve at 500 r.p.m. = $5.9 \times 10^5$ dyne-cm.
$T_2$ = Torque from intercept of extrapolated straight line portion of downcurve = $1.6 \times 10^5$ dyne-cm.

$$N_{500} = \frac{9.55\ T_{500}\ S}{\text{r.p.m.}} = \text{apparent viscosity at 500 r.p.m. in poises}$$

$$= \frac{9.55 \times 5.9 \times 10^5 \times 0.0003262}{500} = 3.66 \text{ poises}$$

$$U_{1000} = \frac{9.55\ (T_{1000} - T_2)}{\text{r.p.m.}}\ S = \text{plastic viscosity at 1000 r.p.m. in poises}$$

$$= \frac{9.55 \times (8.9 \times 10^5 - 1.6 \times 10^5) \times 0.0003262}{1000} = 2.27 \text{ poises}$$

$$U_{500} = \frac{9.55\ (T_{500} - T_2)}{\text{r.p.m.}}\ S = \text{plastic viscosity at 500 r.p.m. in poises}$$

$$= \frac{9.55\ (5.9 \times 10^5 - 1.6 \times 10^5) \times 0.0003262}{500} = 2.67 \text{ poises}$$

$F = T_2C$ = yield stress in g. per sq. cm.
$= 1.6 \times 10^5 \times 0.01465 = 2.3 \times 10^3$ g. per sq. cm.

$$M = \frac{2(U_{500} - U_{1000})}{\ln (1000)^2/(500)^2} = \text{coefficient of shear thixotropy in dyne per cm. sec.}$$

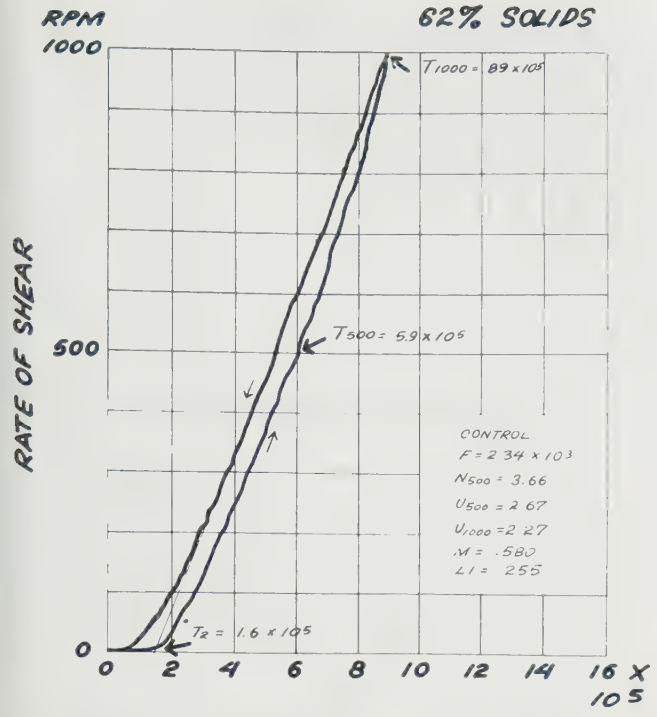


*Fig. 6. Typical starch-latex coating rheogram—62% solids; 85°F.; b bob of Hercules high-shear viscometer*

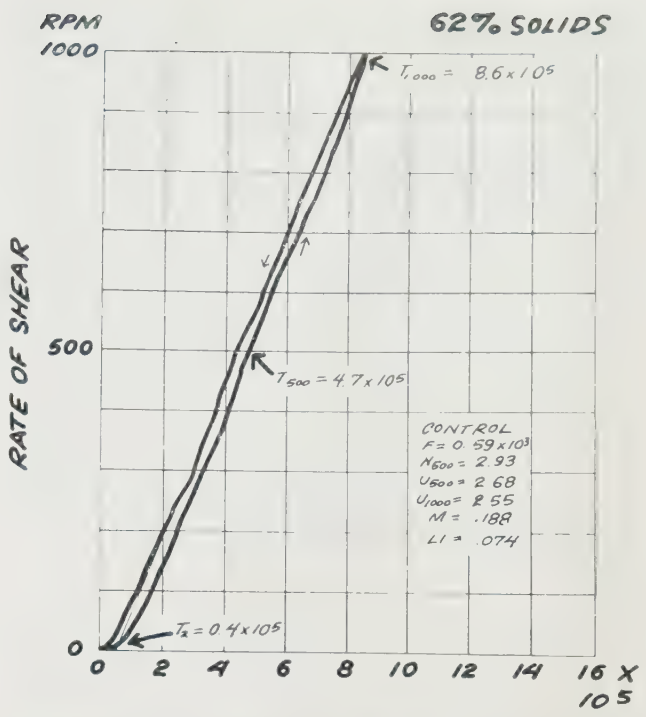


*Fig. 7. Typical protein-latex coating rheogram—85°F.; b bob of Hercules high-shear viscometer*

$$= \frac{2(2.67 - 2.27)}{2 \times 2.3 \times \log 10^2} = 0.580 \text{ dyne per cm.-sec.}$$

$$L.I. = \frac{M}{U_{1000}} = \text{leveling index, dimensionless}$$

$$= \frac{0.580}{2.27} = 0.255$$

Figure 6 shows the starch-latex coating rheogram used for the above calculations.

The term leveling index, a dimensionless ratio of the coefficient of shear thixotropy to the plastic viscosity, is an important measure of the suitability of coating mixtures for high-shear roll coating since it indicates the ease with which the coating will level after leaving the nip (*6*). A high leveling index is obtained if the coating has a high degree of thixotropy and a low plastic viscosity. Normally, the leveling index does not change appreciably with solids content of the coating mixture, since plastic viscosity usually increases proportionally with shear thixotropy as the solids is increased. This indicates that it represents the amount of thixotropy per unit of plastic viscosity inherent in the formulation (*4*). Values for leveling index less than 0.20 are normally considered unsatisfactory, 0.20 to 0.35 indicate borderline performance, and over 0.35 indicate satisfactory performance in high shear roll coatings. Whether or not these values are of significance to high solids trailing blade coatings should be of great importance and will be discussed later on in this paper.

### *Protein Formulation*

According to the literature, most protein and protein-latex high solids coatings exhibit pseudoplastic-thixotropic flow. Thompson and Hansen (*7*) outlined a method of calculating the coefficient of thixotropic breakdown in these systems by taking the difference between the loss of shearing force per unit area per unit increase in rate of shear from the upcurve and the loss of shearing force per unit area per unit increase in the rate of shear from the downcurve.

This is represented by the formula $M' = H - H'$ where $M'$ is the coefficient of shear thixotropy. Therefore a leveling index (*5*) can be calculated from the formula $L.I. = M'/\theta_2$ where $\theta_2$ is the viscosity in

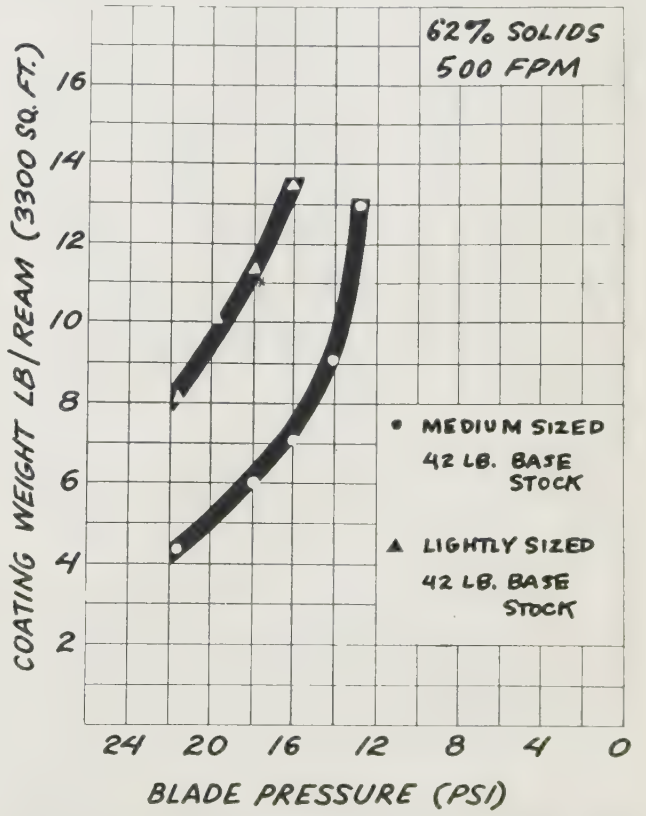


*Fig. 8. Effect of sizing in body stock on coat weight-blade pressure curve—pilot coater at 500 f.p.m.; 62% solids, protein-latex-clay coating; 85°F.; 0.012 in. blade; 42-lb. base stock*

dynes taken from the top of the curve, usually at 1000 r.p.m.

The leveling indices of pseudoplastic-thixotropic systems are normally much lower than those of plastic-thixotropic systems because of the lower amount of thixotropy. However, no published operable limits are available for comparison.

Our protein-latex formulation gave a rheogram that appeared to be more plastic than pseudoplastic with a small yield stress and was slightly thixotropic (Fig. 7). The downcurves were so close to being straight lines that the calculations were made assuming plastic-thixotropic flow.

### *Effect of Body Stock*

Our brief work using body stocks with various degrees of sizing bore out the fact that the more absorbent the body stock, the greater the penetration, and therefore the higher the coat weight at a given blade-pressure setting.

Figure 8 compares coating weight–blade pressure curves of lightly sized raw stock versus medium sized raw stock using the protein-latex formulations at 62% solids. The types of curves shown are typical of those obtained throughout our studies and show that with a given formulation, coat weight increases as the blade pressure is decreased. It will be noted that these plots are nonlinear and show that coating weight increases at a faster rate than blade pressures are decreased. Straight-line relationships can be approximated by plotting the curves on semilogarithmic paper, using the least mean square deviation method.

From the curve plotted in this manner and using the equation:

$$y = mx + b$$

where $m$ = slope of the curve
and $b$ = $y$ intercept

it is possible for a coater to predict the coat weight he can obtain using a given blade pressure setting. We did not attempt to convert the air pressure indicated by the gage from pounds per square inch to pounds per lineal inch at the blade (*1*).

For the major part of our work, we picked a 42 lb. per ream, lightly rosin sized base sheet typical of many publication grades. We also worked briefly with size press treated board. The coatings obtained with size press and prime coated board, as expected, gave lower coat weights at given blade pressure settings than the raw stock because of the lower degree of coating penetration. Although our studies were confined largely to publication grade raw stock, the rheological properties and most of the trends discussed should be applicable to most types of base stocks. However, additional studies with paperboard are warranted and at present are under way.

The body stock is one of the most important variables in any coating operation and cannot be overemphasized. In order to insure that this variable was kept as uniform as possible during our studies, each base sheet was weighed prior to the laboratory coating operation and size tests were run at frequent intervals. In our pilot coater work, raw stock basis weights were taken before and after each run.

## INFLUENCE OF VARIABLES ON FLOW

### *Effect of Per Cent Solids*

As the per cent solids of both our starch and protein formulation was increased all of the flow variables also increased.

Figure 9 compares the rheograms of the starch-latex formulation at 61 and 65% solids. Under the conditions in which our rheological studies were made, we noted that a 4% increase in solids gave about double the value for each rheogram variable.

When applied with the lab coater these same coatings showed that increased solids gave higher coat weights at each blade pressure setting (Fig. 10). This same

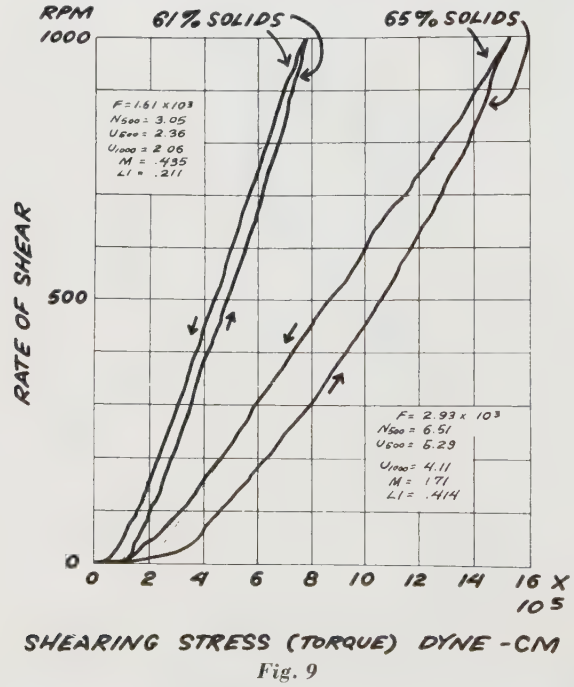


*Fig. 9*

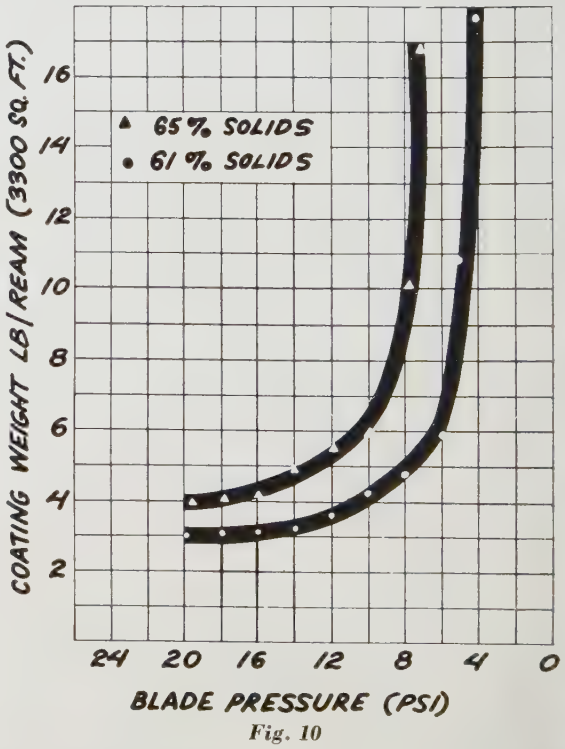


*Fig. 10*

***Effect of per cent solids (Fig. 9) on flow properties (Fig. 10) on coating weight–blade pressure curve—b bob; starch-latex clay coating at 85°F.; lab coater at approx. 500 f.p.m.; 0.012 in. blade; 42-lb. base stock***

effect was true of the protein coatings, but to a lesser degree. It follows, therefore, that increases in one or more of the flow curve values as a result of increasing the solids have produced higher coat weights. Since all of these values changed simultaneously it is difficult at this point to predict which value or values has the dominant influence. For this reason, our further efforts included attempts to study each of these values separately. Practically all our additive work was done at 62% solids, since the starch-latex control without an additive produced a leveling index of approximately 0.25, which for some roll coaters and formulations could be considered as having borderline operability.

### *Effect of Thixotropy and Leveling Index*

As previously discussed, all our coatings were given a 1-hr. standing period prior to running rheograms and applying them. Since the coatings have little chance to be mixed or worked owing to the nature of application in the lab coater, it was decided that an attempt should be made to more closely simulate actual plant conditions where the color is given a good deal of agitation, being pumped to and from the coating pan and worked by the continuous action of the backup cylinder.

A batch of coating was mixed continuously, rheograms run, and coating blade pressure curves plotted. These were then compared with the same coating given a 1-hr. static period before testing. Figure 11 shows that the rheogram for the continuously mixed coating has the same general shape as the static coating. The yield stresses, and plastic viscosity at high shear ($U_{1000}$) are almost identical, however, as expected, the mixing appears to have broken down the gel structure to a certain extent, thus affecting the coefficient of thixotropy and consequently the leveling index.

Again, as expected, when plotting the coat weight-blade pressure curves of these two coatings (Fig. 12) we see that the two are superimposed and the change in leveling index and thixotropy have not affected the coat weight. Further, upon inspecting the coatings, no visual difference in smoothness or pattern could be noticed at any given coat weight. It therefore appears that similar results can be obtained on the laboratory coater whether or not the 1-hr. static period is used. However, the static period allows magnification of some of the rheogram variables so that differences between each additive tested can be more easily detected.

### *Effects of Additives*

As previously stated, most of the additives were applied at the rate of 1% (dry) on the dry pigment in both formulations. If the additive had an extreme effect on the rheological properties, this rate was lessened. Liquid additives were mixed directly into the coating, while paste and dry materials were dispersed first in water, and the final solids adjusted accordingly.

Additives were chosen from such chemical classes as soluble and metallic soaps, fatty mono- and diesters, sulfonated fats and oils, fatty nitrogen compounds, polyglycols, and miscellaneous blends.

Over 50 additives were tested in each of the two coating formulas. Most of the additives tested affected the rheological properties and coating weight–blade pressure curves in one way or another. Although there is no space here to describe them all, we will discuss those which we considered to be most important.

Many of the additives tested increased all the flow variables much the same as the effect of increasing the solids. Figure 13 shows that we can get almost the same rheogram at 62% solids in the starch-latex coating using an additive as we get from a 65% solids coating without the additive (Fig. 9). The resulting blade pressure–coating weight curve (Fig. 14) shows that this additive also increases the coating weight at any blade pressure setting and allows the coat weight to be controlled at higher blade pressures.

Many additives (although not necessarily the same

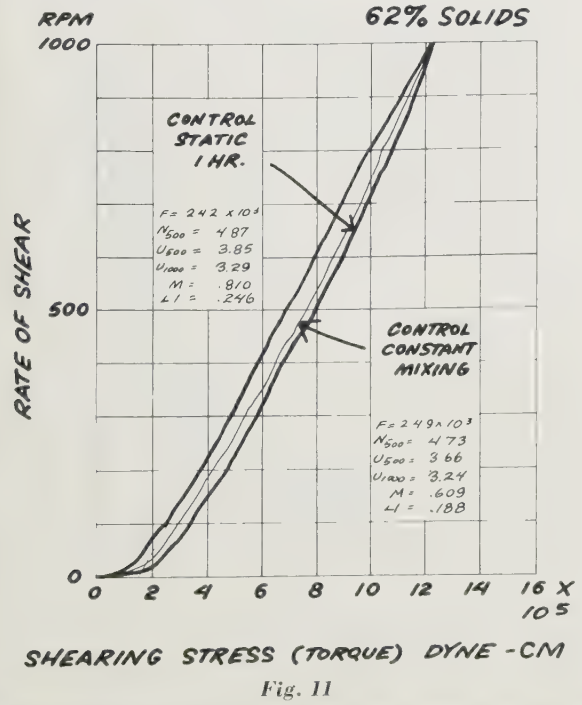


*Fig. 11*

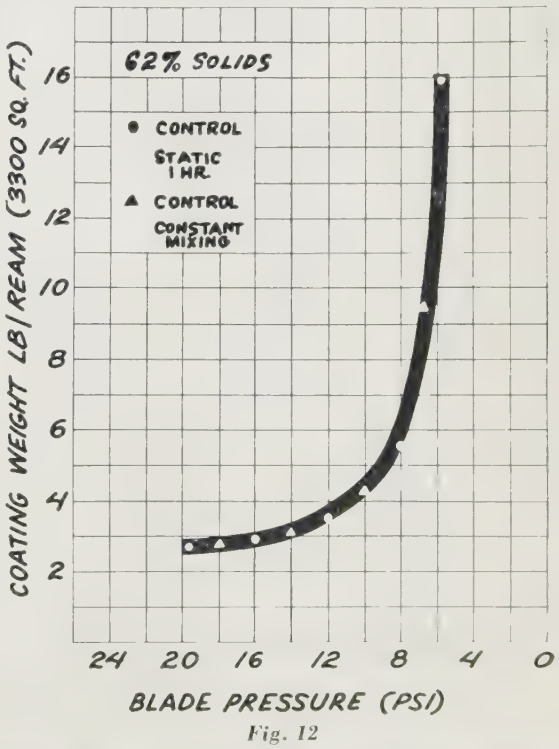


*Fig. 12*

*Effect of constant mixing versus static period (Fig. 11) on flow properties (Fig. 12) on coat weight blade pressure curve 62% solids, starch-latex-clay coating; lab coater at approx. 500 f.p.m., 0.012 in. blade; 42-lb. base stock*

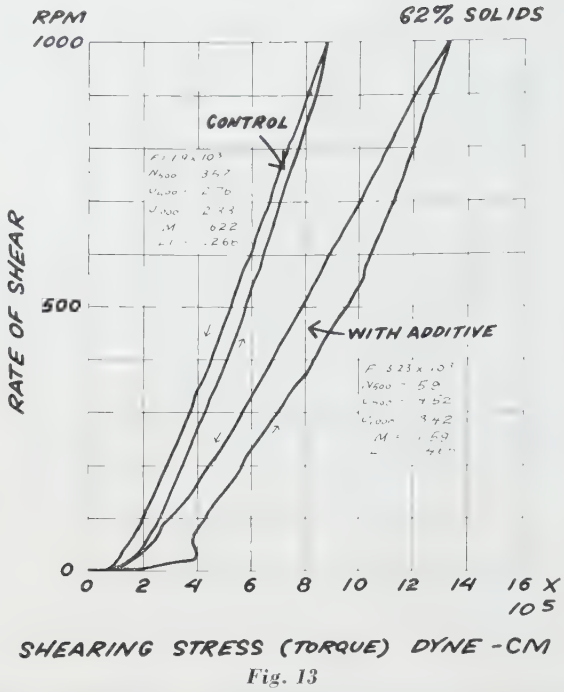


Fig. 13

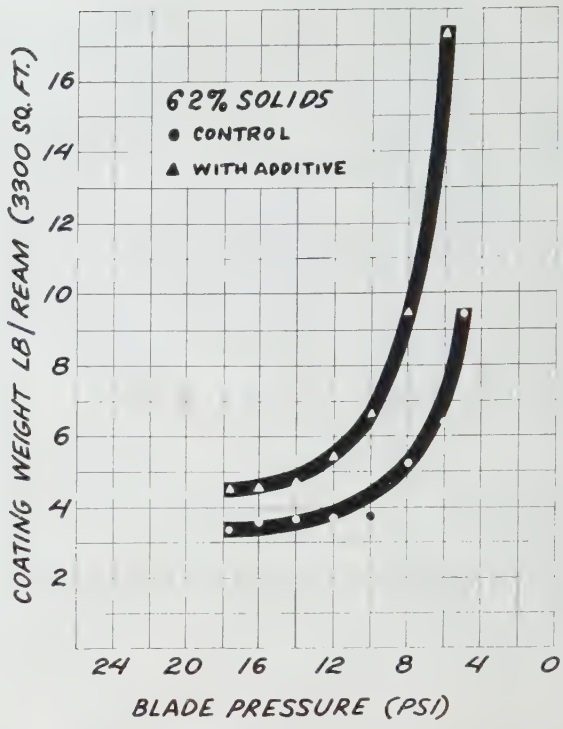


Fig. 14

*Effect of additives; increased (Fig. 13) flow properties (Fig. 14) coat weight–blade pressure curve—62% solids, starch-latex-clay coating; lab coater at approx. 500 f.p.m.; 0.012 in. blade; 42-lb. base stock*

ones) also produced similar effects in the protein-latex formula. This is illustrated by Figs. 15 and 16. Since the viscosity of this coating was quite a bit lower than the starch coating, the control rheogram is more upright and the overall coating weights are lower than those of the starch work. However, once again, higher coat weights were achieved using certain additives. Although the effects are not quite as pronounced in the protein colors as they are in the starch colors, they definitely do occur and are quite reproducible. (There is some variation which may occur in rheograms with the protein formula that can be attributed to a certain amount of entrapped air.) For the purpose of rheogram evaluations, it was felt that care and proper mixing technique to avoid foam was a better procedure than using a defoamer which might in itself affect the slope of the flow curve and the coat-weight curve. Additives which increased coat weight fell into the class of fatty esters, some of the soluble and insoluble soaps, sulfonated oils, and some of the fatty nitrogen compounds. Use of this type of additive may be of great help to the coater who has developed a successful formula for an-

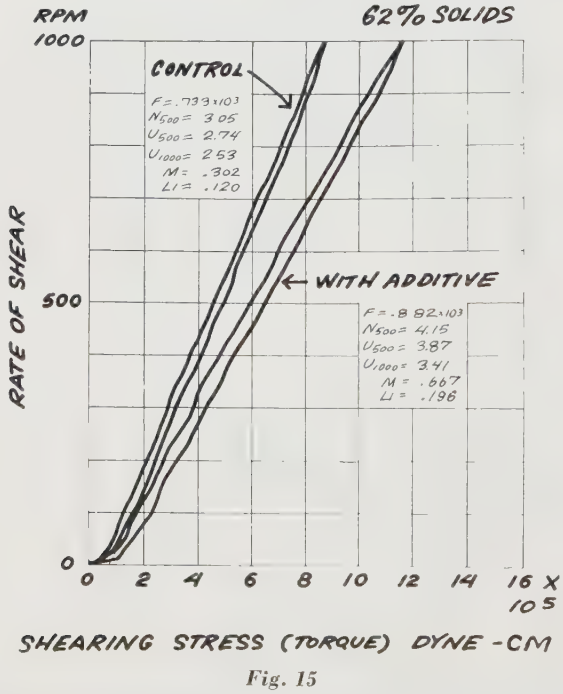


Fig. 15

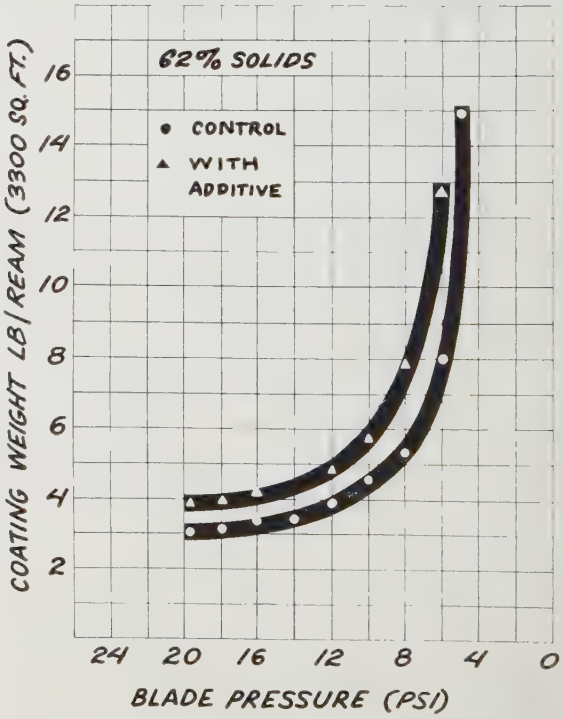


Fig. 16

*Effect of additives; increased (Fig. 15) flow properties (Fig. 16) coat weight–blade pressure curve—62% solids, protein-latex-clay coating; lab coater at approx. 500 f.p.m.; 0.012 in. blade; 42-lb. base stock*

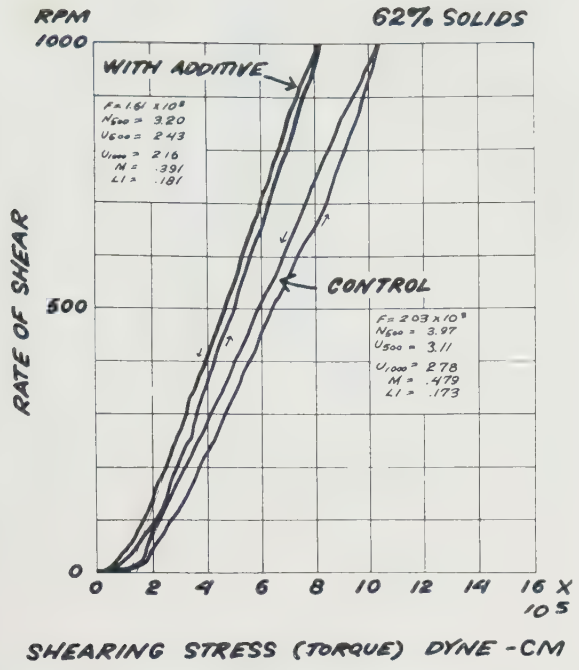


Fig. 17

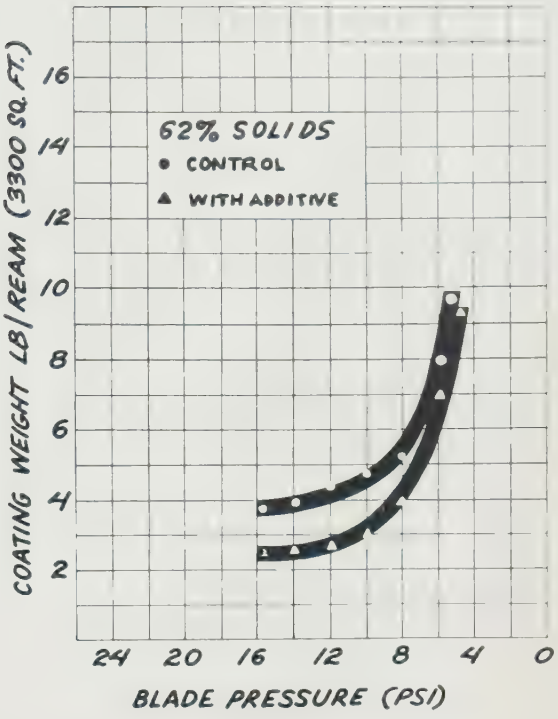


Fig. 18

*Effect of additives; decreased (Fig. 17) flow properties (Fig. 18) coat weight–blade pressure curve – 62% solids, starch-latex-clay coating; lab coater at approx. 500 f.p.m.; 0.012 in. blade; 42-lb. base stock*

other method of coating and wants to use it on a newly installed trailing blade coater needing a viscosity modifier to give him the coat weight he desires.

Such an additive may also aid the coater who has limited dispersing and pumping equipment and cannot increase his solids enough to give him the desired coat weight. A third user may be the mill whose trailing blade coater is limited in controls for adjusting blade pressures to increase his coat weight.

Conversely, several additives in the polyglycol class were found to have just the opposite effect. Figure 17 compares the rheogram of the same starch coating at 62% solids with and without an additive of this type. The additive in this case has lowered the flow values and moved the rheogram to a more upright position. The blade pressure–coat weight curves of these same coatings are compared in Fig. 18, which shows that the additive has lowered the coating weight at all blade-pressure settings. Another additive in this class was found to exhibit the same effects to the protein-latex

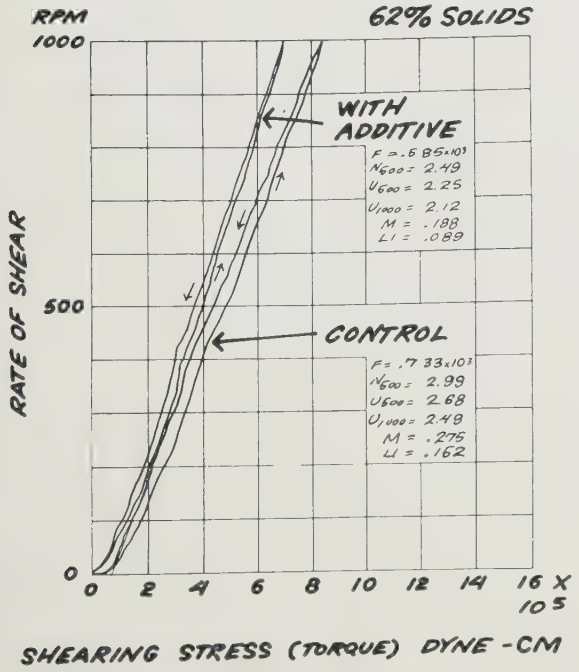


Fig. 19

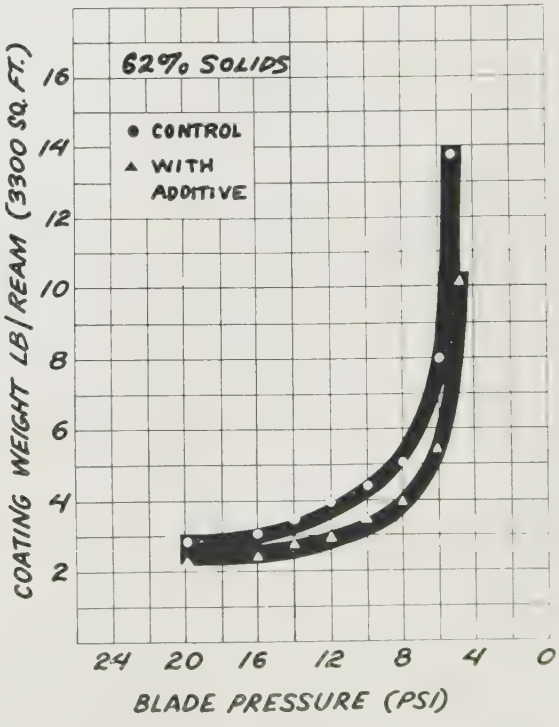


Fig. 20

*Effect of additives; decreased (Fig. 19) flow properties (Fig. 20) coat weight–blade pressure curve – 62% solids, protein-latex-clay coating; lab coater at approx. 500 f.p.m.; 0.012 in. blade; 42-lb. base stock*

coating. This is illustrated by Figs. 19 and 20, and once more proved to us that rheology can be used to predict coating behavior. We further found that by increasing the per cent solids of the coatings which contained additives of this type, we could again obtain about the same coating weight curve as that of a lower per cent solids control. This permits a coater with limited drying capacity to run his coater at higher speeds and obtain the same coat weights at higher solids. Also, it allows the user of a high viscosity coating to go up in solids without adversely affecting his flow properties. This is especially true if a coating is very viscous and can be fluidified by the additive (*8*).

By studying and analyzing the effects of additives, we were able to narrow down further the flow properties which have the greatest effect on coating weight–blade pressure curves. A few of the additives did not greatly affect most of the flow variables, but did increase the yield stress considerably when used in both coating formulations. When the coat weight curve of a coating containing this additive was compared to the same coating without it, it showed slightly higher coat weights at very low blade pressures and about the same coat weights as the control at blade pressures of 8 p.s.i. and higher. From this, we theorized that the force required to overcome the higher yield stress was actually lifting the blade when there were low pressures on it. Also, the shear generated at higher pressures was more than enough to overcome the higher yield stress and therefore the curve assumed the same path as the control. We would expect that blade pressures of less than 8 to 10 p.s.i. would not be used in most trailing blade operations, since at these low pressures the coating weight is out of control in that a slight change in pressure greatly affects coat weight. Therefore, it appears that yield stress is not an important variable as far as coat weight is concerned at normal operating blade pressures. However, yield stress may have some effect if extremely flexible blades are used.

Some of the additives tested increased the coefficient of thixotropy and the leveling index without affecting to any extent the plastic viscosity at high shear ($U_{1000}$). As shown previously with a continuously mixed and a static control, this type of additive had very little effect on the coating weight–blade pressure plot. Here we concluded that any change in the amount of thixotropy, and therefore the leveling index, had little effect on coat weight unless accompanied by a corresponding change in plastic viscosity at high shear. Whether or not this change in thixotropy affected the penetration of the color into the sheet has not been fully investigated. It is believed that a certain amount of viscosity is desirable in order to prevent the coating from penetrating rapidly into the surface of the sheet prior to the shearing action of the blade (depending of course on the absorbency of the base sheet). The shearing action of the blade is so brief that thixotropy and leveling index do not appear to have as much importance in trailing blade coating as they have in transfer roll coating.

All the additives tested which either raised or lowered the coating weight–blade pressure curve were always found to give a corresponding increase or decrease in both plastic and apparent viscosities at the various rates of shear. This was true of both the starch-latex and protein-latex formulations at all percentages of solids which we worked (60–65%).

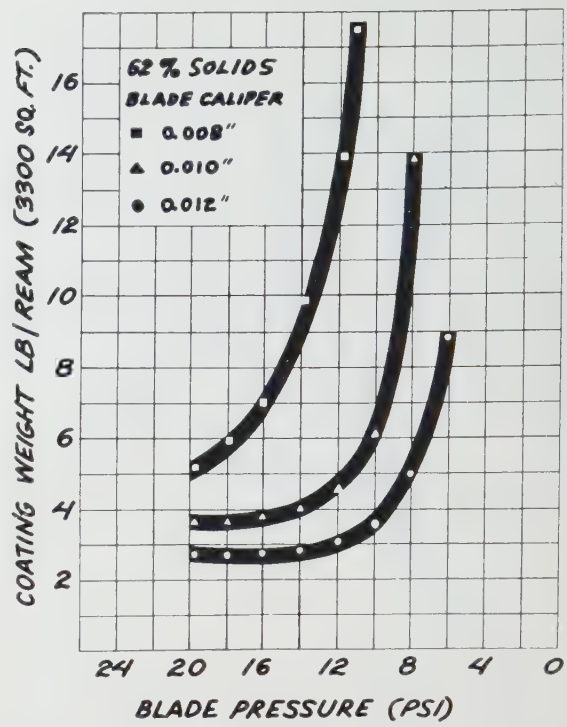


*Fig. 21. Effect of blade caliper or coat weight–blade pressure curve—62% solids, starch-latex-clay coating; lab coater at approx. 500 f.p.m.; 42-lb. base stock*

It should be worthy of mention here that the reproducibility of both rheograms and coat weight was excellent throughout our work. Batch to batch differences shown by the rheograms were almost always traced to inability to reach exactly the calculated solids of our formulations, whereas differences in coat weights from those predicted by the viscometer were usually traced to differences in sizing or basis weight of the raw stock.

### *Blade Caliper and Other Effects*

Throughout our studies we standardized on the 0.012-in. blade in the laboratory coater. To determine the effect of other blade calipers, we studied the coating weight–blade pressure relationships using blades of 0.008, 0.010, and 0.012 in. with the same starch-latex coating. The results, illustrated in Fig. 21, show that the lower the blade caliper and more flexible the blade, the higher the coat weight at each blade-pressure setting. The importance of this study is recognized, since the coater many times must change blades during operation, and has to be sure that the new blade is of the same caliper as the one replaced in order to achieve the same coat weight. Since most mills hone-in their blades before use, so that scratches and streaks from an imperfect blade are minimized, another 0.012-in. blade was honed and run along with the other blades in the series. The honing was made to a 41° angle on the blade (the same angle which the blade makes with the vertical tangent to the backup cylinder of the laboratory coater). This however, had no effect on the coat weight, since the curve made with the honed blade followed exactly that of the unhoned blade.

The effect of temperature on rheograms and coat weights was studied briefly. It was noted that higher temperatures give more fluid coatings and consequently lower the coat weight–blade pressure curves. Our

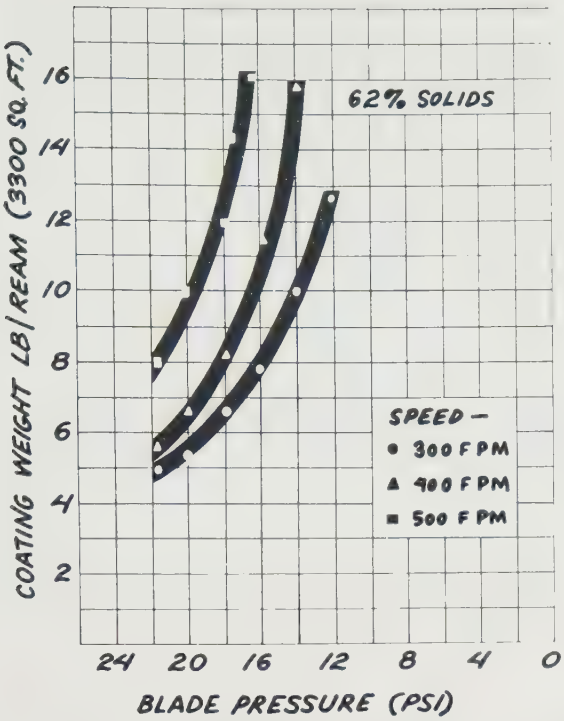


*Fig. 22. Effect of coater speed on coat weight–blade pressure curve—62% solids, starch-latex-clay coating; pilot coater; 0.012 in. blade; 42-lb. base stock*

efforts, as far as temperature was concerned, were confined largely to keeping it as constant as possible so that it could not be considered a variable in our work. Other variables such as blade angle and coater speed as functions of coat weight will be discussed under the pilot coater series.

## PILOT COATER STUDIES

### *Equipment and Coating Preparation*

The pilot coater at the Georgia Kaolin research department in Elizabeth, N. J., proved to be ideal for our pilot studies, since their trailing blade unit is of the same basic design as our modified laboratory coater.

This pilot coater also consists of a rubber backing cylinder fitted with an air pressure controlled 0.012-in. blade in the "puddle" position. The drying equipment contains two infrared heaters, supplemented by two gas-operated heaters. It is designed to operate at speeds ranging from 0 to 750 f.p.m. However, the limited drying equipment restricted the application of higher coat weights to 500 f.p.m.

The same starch-latex and protein-latex formulations were used in these studies as those used in our laboratory work. Master batches were made up at 62% solids in a Kady mill, cooking both pigment and adhesive together, and adding the latex at the end of the mixing cycle. Smaller batches were split up and the various additives mixed in with a low speed agitator. The resultant coatings were very similar in viscosity, and produced almost identical rheograms as those prepared by the laboratory sigma blade mixer (*9*). Brookfield viscosities of the starch-latex coating controls averaged about 45,000 cp. at 10 r.p.m., while those of the protein-latex controls about 5000 cp. at 10 r.p.m.

### *Effect of Coater Speed*

In order to compare the effect of coating speed on the coating weight-blade pressure relationship, coatings were made at 300, 400, and 500 f.p.m. over a wide range of blade pressure settings. The results of these comparisons are illustrated by Fig. 22, which shows the pronounced, effect that higher speed has in increasing the hydrodynamic pressure and producing higher coat weights at any given blade-pressure setting.

This comparison was made with the starch-latex formula at 62% solids. A similar series run with the protein-latex coating produced the same effects, except to a lesser extent.

In general, all the effects noted and previously discussed under Laboratory Studies were verified with the pilot studies. Agents which the laboratory work had indicated would either raise or lower the coating weight–blade pressure curve, also had the same effect on coatings produced with the pilot coater.

The only discrepancy noted between the two studies was that the pilot coater gave much higher coat weight than the laboratory coater at the same blade pressure settings. Since the types of equipment were very similar and the blade caliper (0.012 in.) was the same, other variables were explored.

It was found that the raw stock, although it was acquired from the same mill, and of the same basis weight, gave a much lower size test than the raw stock used in the laboratory studies.

Also, the angle which the blade of the pilot coater makes with the vertical tangent of the backup cylinder was found to be 39°. Some mills which use blade angle to control coat weight have found that as this angle is reduced, the coat weight is increased. Moreover, the blade extension from its fixed point and overhang from the pressure tube are greater on the pilot coater than they are with the laboratory coater. This gives the pilot blade much more flexibility and allows it to be pushed back more easily. Finally, the backup cylinder in the pilot unit is on a movable axis which allows it to be set against the blade or "zeroed in" at 0 p.s.i. Therefore, all the pilot blade pressures recorded were p.s.i.g., and may be actually lower if the pressure needed to fill up the tube had been considered. All these variables between the two types of equipment compounded in the production of higher coat weights at the same speed using the pilot coater. They are summarized in Table II. In order to duplicate the pilot coater blade pressure–coating weight curves with the laboratory model, assuming that all the other variables that can be controlled are the same, probably a lower caliper blade would have to be employed to give the same flexibility.

**Table II. Variables Between Pilot and Laboratory Coaters**

| *Machine* | *Blade angle with vertical tangent* | *Blade extension from fixed point, in.* | *Blade overhang from tube, in.* | *Backup cylinder diameter, in.* | *Blade gage, in.* | *Raw stock (TAPPI Cobb —test-30 sec.)— Wire side, mg./cm.²* | *Felt side, mg./cm.²* |
|---|---|---|---|---|---|---|---|
| Pilot | 39° | 3/4 | 3 3/8 | 18 | 0.012 | 6.2 | 7.1 |
| Laboratory | 41° | 5/8 | 1 13/16 | 7 | 0.012 | 4.1 | 2.5 |

### *Effect of Calcium Stearate Dispersions*

Probably the most effective and widely used additive in trailing blade, as well as most of the other coating applications, is calcium stearate. The fact that calcium stearate dispersions are so widely used for the lubrica-

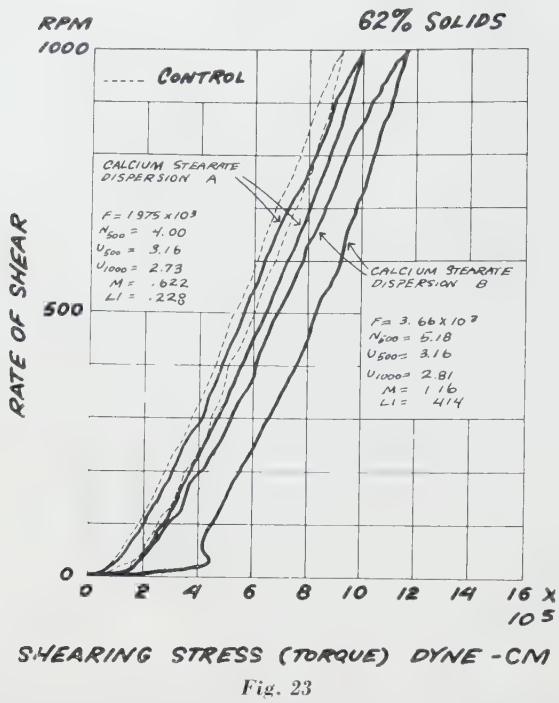


*Fig. 23*

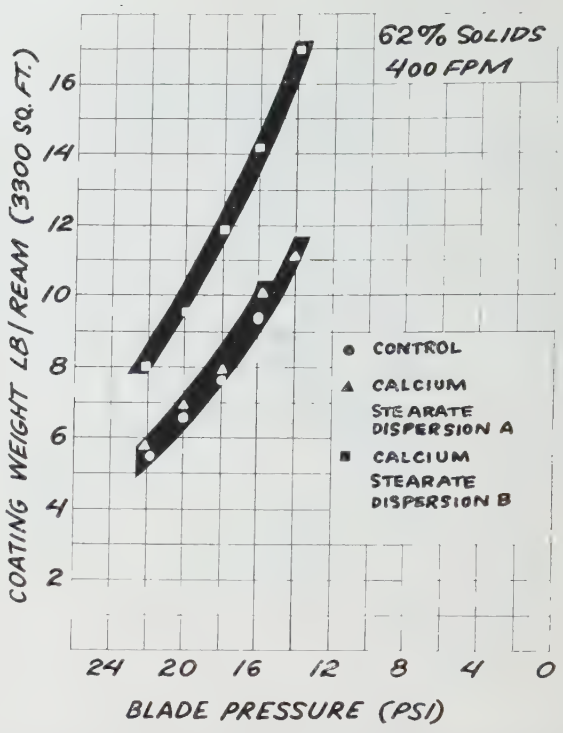


*Fig. 24*

***Effect of calcium stearate dispersions on (Fig. 23) flow properties (Fig. 24) coat weight blade pressure curve –62% solids, starch-latex-clay coating; pilot coater at 400 f.p.m.; 0.012 in. blade, 42-lb. base stock***

tion, ease of calendering, gloss, antidusting, and other surface effects which they perform, make them a logical candidate for rheological study. Our laboratory studies indicated that the type and extent of effect varies with the calcium stearate dispersion. This was verified with the pilot coater studies and illustrated by Figs. 23 and 24. As is shown, some calcium stearate dispersions (Dispersion A) have very little effect on the coating rheogram and the blade pressure–coating weight curve, while others (Dispersion B) greatly increase the flow variables and coat weights at the various blade pressure settings. A third type (not shown) would fall somewhere in between the two extreme illustrated. All these types of calcium stearates are available to the coater. The advantages of one over another are obvious from the previous discussion. The choice is up to the individual mill or application involved and the flow characteristics desired.

### *Surface Effects of Additives*

All the pilot coated papers were supercalendered and given tests for printability (Vandercook), gloss (Ingersoll), pick, brightness, ink holdout, and pattern. Summarized briefly, the printability, pick, brightness, and gloss all increased with increased coat weight. The pick tests were slightly lower with the protein coatings, probably due to the lower adhesive ratio of this color. No visual differences in printability with the various additives could be noted other than improved printing effects with increased coat weight. The printability at 7 to 8 lb./ream (3300 sq. ft.) was considered to be very good. Some of the additives, especially the calcium stearates, showed improved gloss at the same coat weight. This effect was more pronounced with the protein color than with the starch color. Visual inspection under black, light [Ultraviolet high pressure mercury arc lamp (100 W.). Gates Raymaster Black Light] indicated that there was very little pattern with any of the coated papers produced.

Ink penetration appeared to be increased with increased coat weight. Coating penetration appeared to be greater with the protein color, probably because of its lower viscosity and vehicle retention characteristics. None of the additives tested appeared to affect adversely any of the surface or printing properties of the coatings.

It has been found that many trailing blade coatings require a considerable amount of screening to minimize scratches and streaks in the finished sheet. This could be shown quite dramatically in our studies, using a screened coating. The screening operation however, causes entrapped air that may affect the surface of the coated sheet. This problem can be solved by use of the proper defoaming agent. Care should also be taken to choose a defoamer which has a favorable affect on flow properties of the particular coating system.

## SUMMARY AND CONCLUSIONS

A modification of the Time, Inc. bench model trailing blade coater has been developed, allowing laboratory studies of coating variables which correlate extremely well with pilot operation. Rheology studies using the high shear viscometer can be employed in trailing blade investigations to understand how a color will perform on the coater. Together, the results of the modified laboratory coater investigations and high-shear flow property studies can be used to predict how a coating will perform on larger trailing blade equipment. Many of the variables which alter flow properties and coater operation have been investigated. Additives can be employed to perform a major role in controlling flow properties, and their subsequent effect on coating weight and other trailing blade coating properties. No one additive can be looked upon by the coating operator as a

cure-all for all of his problems. Selection of an additive, like that of the pigment-adhesive system, depends upon the flow properties and other effects desired.

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# The Measurement of Relative Molecular Weight of Lignosulfonates by Diffusion

## *III. The Effect of Molecular Size on Surface Active and Other Properties of Lignosulfonates*


JULIUS BENKO



**A new method for determination of relative molecular weight of soluble lignin compounds based on diffusion constants has been previously described by the author (*1, 2*). Employment of this method over a period of several years has made possible a closer evaluation of the effect which molecular size of lignosulfonates has upon certain of the surface active properties of the lignosulfonates. The relationship of the molecular size or weight of lignosulfonic acids to their dispersing properties has been studied for lignosulfonates obtained from a full acid sulfite cook, from a less acid magnefite cook, and from a neutral sulfite semichemical cook. Data are presented correlating molecular size with dispersing effect upon kaolin clays. In general, the dispersing property increases with molecular weight up to an optimum value which may vary with liquors of different origin; but polymerization beyond the optimum molecular weight is shown to be detrimental to this property. On the lignosulfonates from the acid sulfite cook the effect of oxidation and the effect of salt formation with ferrous, ferric and chromic cations upon the dispersing properties is briefly discussed. In addition, the relationship of the relative molecular weight or size to viscosity, surface tension, compressive strength in binders, soil-sealing properties, and to the use of lignosulfonate materials as extenders for phenol-formaldehyde resins is presented.**


Over the years many data have been collected in our laboratories to the effect that the dispersing properties of the high-molecular-weight fractions of lignosulfonates are better than the dispersing properties of either the low-molecular-weight fractions or of the unfractionated spent sulfite liquors. This relationship has been basically the same, regardless of the method used for the fractionation of the spent sulfite liquor. Previously, lacking a method for the estimation of the molecular weight or size, we have had to accept a loosely defined estimation of the molecular size, which was based upon the knowledge that the manner or method of fractionation could be expected to yield lignosulfonates of larger or smaller size classifications.

As soon as the diffusivity method for the determination of relative molecular weight values was developed and relative molecular weight values were established for a number of products developed or fractionated in our laboratories over a period of several years, we were able to confirm the earlier findings.

Analysis of a few commercially available dispersing agents from lignosulfonates has recently shown a similar picture to that for the laboratory products. All products analyzed in this connection have shown that they consist either of the high-molecular-weight fraction isolated from a spent sulfite liquor, or that they have been produced through the polymerization of the whole spent sulfite liquor, in one manner or another, to a comparably high-molecular-weight product.

Polymerization of lignosulfonate products may be carried out in many ways; heat treatment under refluxing without chemical additives is reproducible and especially suitable for laboratory studies. Changes in dispersing properties with polymerization can be readily followed with controlled studies of this nature. It has been established on a number of different liquors that an increase of the relative molecular weight value, in a given range, results in better dispersing properties; but it is also apparent that excessive molecular weight increases are undesirable.

It was decided to present the correlation of the relative molecular weight values and dispersing properties using a number of different molecular-weight polymers of three basically different lignosulfonic acid materials. These are lignosulfonic acids from an acid sulfite cook, from a magnefite cook, and from a neutral sulfite semichemical cook.* An alternative way would be to present a number of data measured previously on lignosulfonate materials of different origin and pretreatments. Experience has shown conclusively that presentation of such data would result in a picture very similar to the one presented in this paper. Presentation of such data, however, would meet many objections to


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* The pulpwood used in producing the spent liquors for which data are reported in this paper was predominantly hardwood (aspen); but other and extensive data indicate that softwood spent sulfite liquors provide closely analogous results.

the effect that the main differences were not due to the molecular size, but rather to the sort of wood used, the type of cooking process, the manner of fractionation, or to the effect of other treatments of the spent sulfite liquors. We admit that such objections are justified, insofar as the above-mentioned conditions are potentially important factors in the obtaining of a particular molecular size and type of lignosulfonate. For tabulating results and comparing data, however, we feel that it is much easier to use just the relative molecular weight figures of samples derived from the same original lignosulfonate material, without the usual lengthy description of the particular lignosulfonates, about which we still lack basic and very important information, namely, the molecular weight or size of the material.

In addition to the discussion of the relative molecular weight and surface active properties relationship, a brief discussion of the relationships of other important properties to the relative molecular weight values will be presented. On different relative-molecular-weight polymers prepared from the lignosulfonates from the acid sulfite cook, the relationship of the relative molecular weight to viscosity, surface tension, compressive strength in binders, soil sealing properties, and to the use of lignosulfonate polymers as an extender for phenol-formaldehyde resins is presented.

## EXPERIMENTAL

### *Preparation of Samples*

Substantially free lignosulfonic acids containing only low percentages of base cations were prepared by means of electrodialysis from two spent sulfite liquors, and by means of partial cation exchange on an Amberlite IR-120 ion-exchange resin bed from a third liquor. Since electrodialysis removes not only base cations from the treated spent sulfite liquor, but also the low-molecular-weight components of the liquor, the liquor which has been base-ion depleted by means of cation exchange has been fractionated prior to that step by means of dialysis in a Brosites cell for several days.

Table I gives a brief analytical description of these samples, from which the different relative molecular weight subsamples were prepared by polymerization.

Polymers of different relative molecular weights were prepared in all but three of the reported cases by refluxing at atmospheric pressure for appropriate periods of time. From the electrodialyzed ammonia-base sulfite liquor, three additional samples were prepared by means of spray-drying. Analytical data on the subsamples are presented in Tables II, III and IV, following the brief description of the analytical procedures used in the tables.

**Table I. Analytical Data of the Samples**

| *Sample no.* | *Description of material* | *MeO, %* | *S, %* | *Equiv. wt.* | *$H^+/S$* | *Rel. MW*[a] |
|---|---|---|---|---|---|---|
| 1 | Electrodialyzed ammonia-base acid sulfite liquor | 8.20 | 7.05 | 262 | 1.60 | 6300 |
| 2 | Electrodialyzed neutral sulfite semichemical liquor | 6.30 | 9.84 | 166 | 1.73 | 1740 |
| 3 | Magnefite liquor, dialyzed in a Brosites cell, then partially base-ion depleted by means of ion exchange | 10.74 | 7.05 | 437.5 | 1.22 | 9100 |

[a] Relative molecular weight values were measured following fractionation by means of ion exclusion.

### *Determination of the Relative Molecular Weight of Lignosulfonates By Diffusivity Measurements*

Sodium salts of the lignosulfonic acids, fractionated by means of ion exclusion or by means of exhaustive dialysis through cellophane, were dissolved in a strong electrolyte solution and diffused into such an electrolyte solution through a medium porosity, fritted glass disk. For the electrolyte solution a 0.1*N* KCl solution is used; the amount of diffusate and that of the undiffused material is established by means of optical density readings on a Beckman DU spectrophotometer at 281.5 m$\mu$. The diffusivity rate of the lignosulfonate material is then related to that of the substance used for the calibration of the diffusion cells and the relative molecular weight of the lignosulfonate material is calculated from the equation:

$$MW_1 = \left(\frac{\lambda_2}{\lambda_1}\right)^2 \times MW_2 \qquad (1)$$

A detailed description of the determination of relative molecular weight values for lignosulfonates was presented earlier in this series (*1*, *2*).

### *Measurement of the Dispersing Effect of Lignosulfonates*

The dispersing property of lignosulfonates is evaluated on a kaolin-water slurry prepared from 100 g. of kaolin, 76 ml. of distilled water and 74 ml. of saturated lime water in such a way as to obtain a slurry of pH 7.0. Slight variation of the water–lime water ratio is sometimes necessary to obtain the exact pH. The viscosity of the slurry is measured using a Brook-

**Table II. Summary of Dispersion Test Results and Relative Molecular Weight Data on Three Series of Polymerized Lignosulfonate Materials**

| *Electrodialyzed ammonia-base acid sulfite liquor* | | | | *Electrodialyzed neutral sulfite Semichemical Liquor* | | | | *Dialyzed Magnefite liquor, base-ion depleted by ion exchange* | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| *Sample no.* | *Rel. MW*[a] | *Viscosity of slurry, cp.* | *Additive used, ml.* | *Sample no.* | *Rel. MW*[a] | *Viscosity of slurry, cp.* | *Additive used, ml.* | *Sample no.* | *Rel. MW*[a] | *Viscosity of slurry, cp.* | *Additive used, ml.* |
| 1 | 6,300 | 1675 | 5.0 | 1 | 1,740 | 160 | 4.5 | 1 | 9,100 | 1670 | 5.0 |
| 2 | 8,100 | 680 | 5.0 | 2 | 3,490 | 140 | 4.0 | 2 | 13,300 | 125 | 3.15 |
| 3 | 8,900 | 450 | 5.0 | 3 | 4,020 | 160 | 3.5 | 3 | 18,200 | <50 | 2.45 |
| 4 | 10,900 | 200 | 5.0 | 4 | 4,490 | 150 | 3.4 | 4 | 22,200 | <50 | 2.30 |
| 5 | 12,050 | 110 | 5.0 | 5 | 5,080 | 100 | 3.4 | 5 | 39,300 | 50 | 2.80 |
| 6 | 14,750 | 45 | 4.5 | 6 | 5,560 | 100 | 3.6 | 6 | 54,800 | 100 | 3.25 |
| 7 | 16,800 | 45 | 4.4 | 7 | 7,280 | 100 | 3.6 | 7 | 56,500 | 100 | 3.78 |
| 8 | 20,000 | 45 | 4.5 | 8 | 8,000 | 100 | 3.6 | | | | |
| 9 | 21,200 | 65 | 4.2 | 9 | 8,550 | 100 | 3.6 | | | | |
| 10 | 21,700 | 60 | 4.7 | 10 | 8,880 | 100 | 3.6 | | | | |
| 11 | 24,000 | 100 | 5.0 | 11 | 10,700 | 100 | 4.1 | | | | |
| 12 | 26,400 | 250 | 5.0 | 12 | 12,900 | 100 | 4.2 | | | | |
| 13 | 28,200 | 335 | 5.0 | 13 | 16,700 | 100 | 4.4 | | | | |
| 14 | 30,300 | 600 | 5.0 | | | | | | | | |
| 15 | 42,000 | 975 | 5.0 | | | | | | | | |

[a] Determined following ion exclusion.

field viscometer, and its value prior to the use of additives is about 5000 cp.

The slurry is then titrated with a 10% sodium lignosulfonate solution of pH 7.0 until the viscosity of the slurry is reduced to 100 cp. or less, or until 5 ml. of the lignosulfonate solution is added. The test, as described above, is considered to be useful as a general screening test. It should be emphasized that results under actual field conditions are not necessarily the same as obtained by the above testing method, since many of the actual conditions during application may greatly differ from those outlined above.

## RESULTS

### *Variations in Dispersing Properties of Lignosulfonic Acids with Relative Molecular Weight*

Several subsamples of different relative molecular weight were prepared from each of the samples described in Table I by means of refluxing under atmospheric pressure for different lengths of time. All subsamples, as well as the samples prior to polymerization, were tested for dispersing properties and for relative-molecular-weight values. All related data are presented in Table II.

*Comments.* Data for each of the series in Table II show that the dispersing properties of lignosulfonic acids underwent characteristic changes with the progress of the polymerization. Polymerization first results in gradual improvements of the dispersing properties, then a molecular weight range for optimum dispersing action is obtained, and finally, polymerization above this optimum molecular weight range results in gradual deterioration of the dispersing properties.

The molecular weight range in which optimum dispersing action is obtained is different for the different liquors. The lowest relative molecular weight range with optimum dispersing properties is established on the polymers derived from the electrodialyzed neutral sulfite semichemical liquor. The optimum relative molecular weight range for dispersing properties is much higher for the electrodialyzed ammonia-base acid sulfite liquors, and it is highest for the polymers derived from the magnefite liquor.

The order of the increasing equivalent weights of these three liquors, as shown in Table I, reflects the same pattern as the order of their optimum molecular weight ranges for best dispersing properties. While this might be purely accidental, it is suggested that this relationship should be noted.

In preparing different relative molecular weight materials from the same lignosulfonic acid sample by means of polymerization, it has been repeatedly established that analytical data on lignosulfonic acids indicate little or no changes during the progress of polymerization. Polymerization by its nature is a macromolecular rearrangement of the lignosulfonic acids, involving little or no changes of the methoxyl and sulfur percentages and of the equivalent weight values. This is shown in Table III on a few samples derived from the electrodialyzed, ammonia-base, spent sulfite liquor.

The variation of the values presented in Table III, except for the relative molecular weights, is too small to explain changes connected with the different levels of polymerization.

**Table III. Chemical Analysis of Samples**

| *Sample no. in Table II* | *Rel. MW following ion-exclusion* | *MeO, %* | *S, %* | *Equiv. wt.* |
|---|---|---|---|---|
| 1 | 6,300 | 8.21 | 7.05 | 262 |
| 5 | 12,050 | 8.16 | 7.07 | 264 |
| 9 | 21,200 | 8.45 | 6.94 | 267 |
| 14 | 30,300 | 8.37 | 6.81 | 263 |
| 15 | 42,000 | 8.37 | 7.05 | 260 |

### *Variations in Dispersing Properties of Spray-Dried Lignosulfonic Acids with Relative Molecular Weight*

Spray-dried solids have been prepared from the electrodialyzed ammonia-base acid sulfite liquor under three sets of conditions. Relative molecular weights of the products were established following exhaustive dialysis through cellophane and the dispersing properties were measured on the kaolin slurry. Experimental data are presented in Table IV.

**Table IV. Variation of Relative Molecular Weights and Dispersing Properties with the Spray-Drying Conditions**

| *Sample no.* | *Rel. MW on samples fractionated by exhaustive dialysis* | *Weight % in the fractionated sample* | *Viscosity of kaolin slurry, cp.* | *Additive used, ml.* |
|---|---|---|---|---|
| A | 9,760 | 31.8 | 825 | 5.0 |
| B | 16,300 | 46.7 | 125 | 5.0 |
| C | 16,500 | 53.7 | 50 | 5.0 |

Data presented in Table IV show that by varying the spray-drying conditions, the extent of polymerization achieved varies within wide limits; dispersing properties are shown to follow the extent of polymerization. Relative molecular weight data for the spray-dried products were established on samples purified by exhaustive dialysis through cellophane rather than on samples fractionated by means of ion exclusion. This is done because ion exclusion does not always provide a good fractionation for spray-dried products.

### *Effect of Oxidation on the Dispersing Properties of Lignosulfonates*

Oxidation of free lignosulfonic acids with hydrogen peroxide results in an increase of the acidic groups on the lignosulfonate material and also in an improvement of the dispersing properties.

An aliquot of the electrodialyzed ammonia-base acid sulfite liquor described in Table I has been oxidized with $H_2O_2$ at 80°C., using one volume of concentrated reagent for ten volumes of 50% solid containing lignosulfonic acids. The equivalent weight of the oxidized product became 204, as compared with 262 prior to this treatment. A few polymers prepared from the electrodialyzed, ammonia-base, acid sulfite liquor (described in Table II) were also oxidized under similar conditions, and a comparison of the viscosity values measured on the kaolin slurry prior to and following the

**Table V. Effect of Oxidation with Hydrogen Peroxide on Dispersing Properties of Lignosulfonates**

| *Sample no.* | *Rel. MW (ion-excl.)* | *Prior to oxidation: Viscosity of kaolin slurry, cp.* | *Prior to oxidation: Additive used, ml.* | *Following oxidation: Viscosity of kaolin slurry, cp.* | *Following oxidation: Additive used, ml.* |
|---|---|---|---|---|---|
| 1 | 6,300 | 1675 | 5.0 | 150 | 4.5 |
| 2 | 12,050 | 110 | 5.0 | <100 | 3.5 |
| 3 | 21,200 | 65 | 4.2 | <100 | 4.0 |
| 4 | 42,000 | 975 | 5.0 | <100 | 4.0 |

oxidation of the lignosulfonate additives is presented in Table V.

Table V shows that oxidation with $H_2O_2$ results in a marked improvement of the dispersing properties of lignosulfonic acids, regardless of their relative molecular weight. The oxidation results in an increase of the acidic groups on the lignosulfonate molecule, but under the given conditions change does not involve the macromolecular structure of the lignosulfonic acids. It is suggested that the oxidation takes place on $\gamma$-carbon atoms of the phenylpropane units. Experimental evidence, not given here in detail, has shown that depolymerization to a measurable extent first takes place when using higher than the indicated percentages of $H_2O_2$.

### *Effects of Ferrous, Ferric and Chromic Salts Upon the Dispersing Properties of Lignosulfonic Acids*

For establishing comparative data on the effect of different cations on the dispersing properties of lignosulfonates, ferrous, ferric, chromic and a mixture of ferrous-chromic salts were prepared from the electrodialyzed, ammonia-base, acid sulfite liquor described in Table I. Lignosulfonic acids were mixed with equivalent amounts of the metal hydroxides, based upon titration of the lignosulfonic acids with sodium hydroxide. While equivalent amounts of the different hydroxides were used, the pH values of the polyvalent metal salts were lower than that of the sodium salt. This fact is explained by the weaker basic character of the metal hydroxides, as well as by possible chelating effects.

Ferrous, ferric, chromic and ferrous-chromic salts were also prepared from the oxidized lignosulfonic acids, and all products were spray-dried under identical conditions. Results of the dispersion tests performed on these products are given in Table VI.

These data indicate some improvement of the dispersing properties on the ferric salts, and some deterioration on the ferrous salts. When compared with the sodium salts, the chromium salts of lignosulfonates show little, if any, difference in dispersing properties. The salts prepared from the oxidized lignosulfonic acids always show remarkably better dispersing properties than the salts prepared from the unoxidized lignosulfonates. Relative molecular weight data were established on the different salts using different lengths of dialysis time in the fractionation step. The results presented in Table VII show that the lignosulfonate salts with the polyvalent cations have significantly slower diffusivity rates than the sodium salt of the same lignosulfonate material, and thus result in higher relative molecular weights. Furthermore, a higher percentage of the polyvalent

**Table VI. Effect of Different Cations on Dispersing Properties**

| *Description of sample* | *Unoxidized* | | *Oxidized* | |
|---|---|---|---|---|
| | *Viscosity of kaolin slurry, cp.* | *Additive used, ml.* | *Viscosity of kaolin slurry, cp.* | *Additive used, ml.* |
| Sodium lignosulfonate | 1675 | 5.0 | 150 | 4.5 |
| Ferrous lignosulfonate | 2050 | 5.0 | 1100 | 5.0 |
| Ferric lignosulfonate | 1300 | 5.0 | 150 | 4.5 |
| Chromic lignosulfonate | 1800 | 5.0 | 250 | 5.0 |
| Ferrous-chromic lignosulfonate | 1700 | 5.0 | 700 | 5.0 |

**Table VII. Effects of Different Cations on Dialysis Rates and Relative Molecular Weights**

| *Description of sample* | *Relative molecular weight* | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | *7 days diff.* | | *3 days diff.* | | *15 hr. diff.* | | *6 hr. diff.* | |
| | *MW* | *Wt. %* | *MW* | *Wt. %* | *MW* | *Wt. %* | *MW* | *Wt. %* |
| Sodium salt | 10,050 | 24.5 | 8,650 | 28 | 5440 | 35.6 | 3750 | 43.5 |
| Ferric salt | 12,650 | 28.65 | 12,500 | 29.9 | 7340 | 42.4 | 4580 | 49.4 |
| Ferrous salt | 15,500 | 27 | 12,550 | 29 | 7280 | 41.8 | 4900 | 49.0 |
| Chromic salt | 15,150 | 26.5 | 11,900 | .. | 6640 | 41.7 | 4760 | 48.0 |
| Ferrous-chromic salt | 12,200 | 25.2 | 12,550 | 28 | 5770 | 40.3 | 3760 | 48.8 |

salts remains undiffused through cellophane during identical diffusion periods, particularly in the case of the shorter diffusion periods. These data indicate the possibility of some cross-linking effect between lignosulfonate molecules by the polyvalent metal ions, which gradually decreases in dilute solutions but is still noticeable even after seven days.

### *Effect of Relative Molecular Weight Upon Various Properties of Lignosulfonic Acids*

In addition to the investigation of the relationship of dispersing properties to the relative molecular weights, other important end-use properties of lignosulfonic acids were investigated and also found to be related to the relative molecular weight values.

Polymers prepared from the electrodialyzed, ammonia-base, acid sulfite liquor described in Table I were used in the following studies, in which the relationship of the relative molecular weight to the viscosity and surface tension of aqueous lignosulfonate solutions, compressive strength of binders, soil-sealing properties and the phenol-formaldehyde resin extender properties of lignosulfonates were investigated. Relative molecular weight data, viscosity and surface tension values of the samples used in these studies are presented in Table VIII.

Relative molecular weight values were determined on ion excluded and on cellophane diffused samples. The data in column 5 show the extent to which high-molecular-weight material fails to diffuse through cellophane. When ion exclusion is used for the fractionation of lignosulfonate samples prior to establishing of relative molecular weights, some 70 to 80% of the solids are included in the sample material used for diffusivity measurement. Using exhaustive dialysis through cellophane for fractionation, the weight percentage remaining undiffused, and thus the percentage of the original sample on which diffusivity measurement is performed, varies between wide limits.

The following comments may be made:

1. *Relative Molecular Weights.* These values show a continuous increase for samples 1 to 16. Simultaneously, there is an increase in the weight percentage of the material undiffused through cellophane.

2. *Fractionation of Spray-Dried Products.* Samples 17 to 19 show that ion exclusion does not provide the ideal way of fractionation for some spray-dried products. According to the molecular weight information, as obtained following ion exclusion, depolymerization should have taken place in the case of samples 17 and 18. At the same time all other tests, including relative molecular weights following exhaustive dialysis indicate that polymerization has occurred. Both statements are correct, since apparently a change in the size and character of various fractions of the whole material has occurred in the spray drier. It is indicated that a

Table VIII. Relative Molecular Weights, Viscosities and Surface Tension Values of Lignosulfonates

| Sample no. | Description of materials | Relative molecular weight: Ion excluded | Relative molecular weight: Cellophane diffused | Relative molecular weight: Weight % not diffused | Viscosity, sec. effluent time[a] | Surface tension, dynes/cm. |
|---|---|---|---|---|---|---|
| 1 | Electrodialyzed spent sulfite liquor | 6,300 | 9,500 | 26.8 | 78.9 | ... |
| 2 | Refluxed | 8,100 | ... | ... | 83.45 | 48.9 |
| 3 | Refluxed | 8,900 | 11,300 | 38.25 | 84.15 | ... |
| 4 | Refluxed | 10,900 | ... | ... | 85.4 | 50.0 |
| 5 | Refluxed | 12,050 | 19,500 | 44.60 | 87.85 | ... |
| 6 | Refluxed | 14,750 | ... | ... | 89.3 | 50.9 |
| 7 | Refluxed | 16,800 | 22,800 | 45.25 | 90.9 | ... |
| 8 | Refluxed | 20,000 | ... | ... | 94.75 | 52.9 |
| 9 | Refluxed | 21,200 | 31,200 | 52.7 | 104.6 | 52.9 |
| 10 | Refluxed | 21,700 | ... | ... | 104.0 | ... |
| 11 | Refluxed | 24,000 | 32,900 | 53.5 | 111.8 | ... |
| 12 | Refluxed | 26,400 | ... | ... | 114.25 | 56.6 |
| 13 | Refluxed | 28,200 | ... | ... | 124.65 | ... |
| 14 | Refluxed | 30,300 | 47,800 | 61.4 | 132.65 | 57.8 |
| 15 | Reflnxed | 31,900 | ... | ... | 127.9 | 58.1 |
| 16 | Refluxed | 42,000 | ... | ... | 169.35 | 59.2 |
| 17 | Spray-dried—A | 4,310 | 9,760 | 31.8 | 81.6 | 48.8 |
| 18 | Spray-dried—B | 5,500 | 16,300 | 46.7 | 86.3 | 49.7 |
| 19 | Spray-dried—C | 9,000 | 16,500 | 53.7 | 87.6 | 51.3 |

[a] Effluent time of distilled water under the same conditions was 59.9 sec.

portion of the sample material underwent decomposition or depolymerization, resulting in products which are not well enough separated by ion exclusion. The relative molecular weight of these products is assumed to be in the range 2 to 4000. On the other hand, polymerization has occurred and is indicated by the overall picture.

3. *Viscosities* were measured on lignosulfonate sodium salts at pH 7.0, using an Ostwald viscometer immersed in a constant temperature bath of 29.4°C. Readings were taken at 10, 2 and 0.5% solids, but are shown only for 10% concentration in Table VIII. As Fig. 1 shows, there is a good correlation between the viscosity and relative molecular weight values. The range of the variation, however, is much less in the viscosity values than in the relative molecular weights. Even in the case of 10% solids, the range of variation is about 10 sec. in viscosity readings for the molecular weight range usually present in spent sulfite liquors. The variation naturally becomes much less when viscosities on less diluted solutions are measured. Considering these facts and possible interferences in the viscosity readings by substances which would not interfere with the optical density readings, it seems obvious that diffusivity measurements are better adapted to describe macromolecular changes of lignosulfonates than viscosity readings.

4. *Surface Tension* was measured on a Du Noüy surface tensiometer on 10% lignosulfonate solutions at pH 7.0. The results are presented in Fig. 2, and they indicate a good correlation between relative molecular weight data and the surface tension values.

5. *Ore-Binding Properties.* A few of the materials described in Table I have been tested for ore-binding properties. The increase of the molecular size results in an increase of the ore binding or crushing strength properties. The maximum compressive strength increase of the iron ore pellets was, in the present case, about 67% over that of the original value and shows a straight line, first order pattern with the increase of the relative molecular weight values. Experimental data are shown in Fig. 3.

6. *Extenders for Phenol-Formaldehyde Resins.* A few materials described in Table VIII have been evaluated for use as extenders in phenol-formaldehyde resins. It has been found in these materials, as well as in a number of others, that there is an optimum relative molecular weight range for this purpose at around 20,000. Much below this range difficulties with sticking and blistering are encountered, and much above this range the flow properties of the mixtures decrease considerably.

7. *Soil-Sealing Properties.* Lignosulfonic acids of different molecular weight have been investigated repeatedly for such properties, and the conclusion of that work is that with increasing molecular weight,

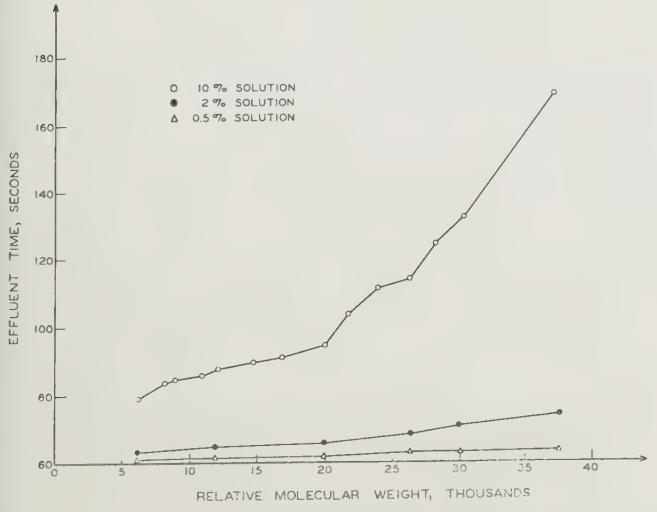


*Fig. 1. Viscosity changes with increasing relative molecular weight*

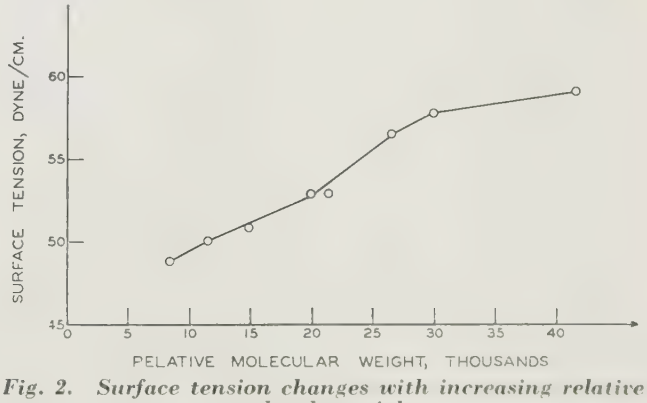


*Fig. 2. Surface tension changes with increasing relative molecular weight*

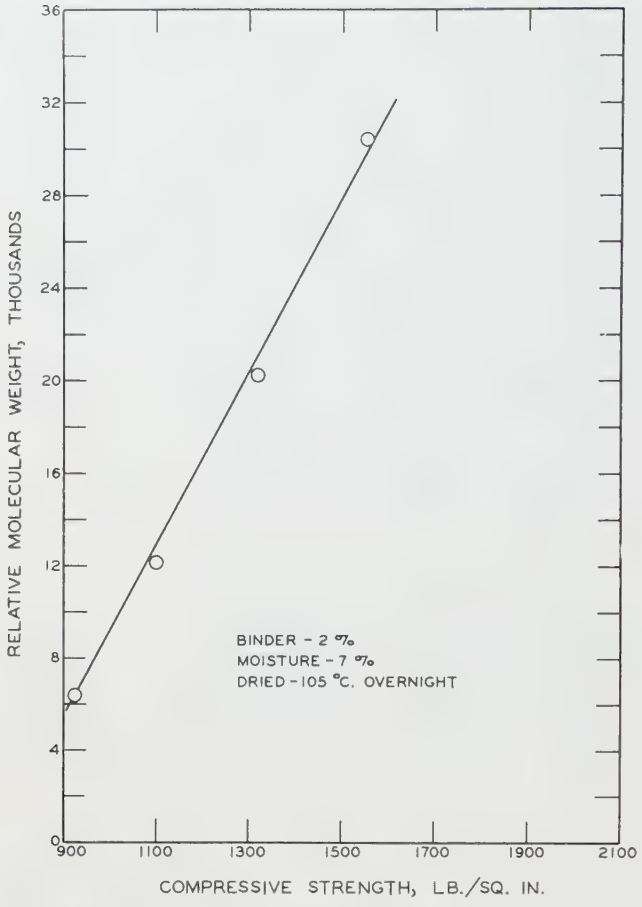


*Fig. 3. Compressive strength–relative molecular weight relationship*

the water-sealing effect of lignosulfonic acids greatly increases. Tests on a few samples from Table VIII have been performed, and the results follow the previously established general pattern.

## SUMMARY AND CONCLUSIONS

1. The molecular weight or size of lignosulfonate materials, as established by a diffusivity method, is shown to have an important effect upon their dispersing properties. In the case of the examples presented, the molecular weight range in which optimum dispersing action is obtained is higher than that obtained during the cook. Therefore, polymerization of the samples results first in improving the dispersing properties, and then in reaching an optimum range. Excessive polymerization is shown to be detrimental. The optimum relative molecular weight ranges are different for lignosulfonate materials of different origin.

2. Other important application properties are also related to the relative molecular weight values. Ore-binding and soil-sealing properties both show continuous improvement with increasing relative molecular weight. For phenol-formaldehyde resin extenders, an optimum molecular weight range is established. Viscosity data and surface tension values of the aqueous solutions of different molecular size lignosulfonates show good correlation with the molecular weight information obtained from diffusivity measurements.

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# The Viscosity and Gelation of Bichromated Gum Arabic Solutions as Affected by Urea

ARTHUR P. HARRISON

**Gum arabic solutions from several commercial sources were sensitized with ammonium bichromate and compared in viscosity change and in time-to-gel. The time-to-gel varied from days to weeks for the different gums, although pH and density were the same for each at the time that identical amounts of the bichromate were added. Urea added to these gums when bichromated delayed the gelation time in every case, but to different degree for each gum. Selective addition of urea modified the gelation time to approximately the same for all. Thus the tendency of various bichromated gum arabic preparations to increase in viscosity and to gel with age can be modified by adding appropriate quantities of urea. The effect of urea is most marked in the more concentrated solutions in the near-neutral range. Urea is nearly neutral itself. The effect of urea is also appreciable in more dilute solutions and in acidic or ammoniated bichromated gum. The effect is to retard or prevent the increase in viscosity and extend gelation time, possibly functioning as an antioxidant.**

The viscosity of coatings of gum arabic is critical for proper coverage under particular conditions of application. Natural gum arabic is most viscous near the neutral range, about 5–8 pH. The viscosity is notably less under increasing acidity or alkalinity. When bichromated sufficiently for practical light sensitivity, gum arabic solutions near the neutral range tend to increase rapidly in viscosity and actually gel in a relatively short time, especially since for practical coating purposes the concentration is more than that critical for setting. When first introduced unammoniated, bichromated gum arabic solutions of 14°Bé. were not recommended for use after a day or so, and the ammoniated solutions were believed unsatisfactory after a month (*4*).

Gum arabic (*3*) is completely soluble in hot or cold

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water, yielding a viscous solution of adhesive paste. It is exuded from points of injury to the bark of the acacia tree as pearllike "tears." Chemically it is the mixed calcium, magnesium, and potassium salt of arabic acid. It is a carbohydrate containing a complicated arrangement of *d*-galactopyranose, *d*-glycuronic acid, l-rhamnopyranose, and *l*-arabofuranose. The molecular weight is about 240,000 and its equivalent weight is about 1100.

Gum arabic tears are graded by size and color, and of course are not all of exactly the same composition. Consequently, their solutions may not have exactly the same characteristics. Solutions from different sources, though of the same density, may differ considerably in viscosity. When available in sufficient variety and quantity, appropriate blending may result in a product of desirable characteristics. On the other hand, appropriate adjustment with urea may be satisfactory.

Gums from eight different suppliers were bichromated in our laboratory in the same way and at the same time. They gelled at intervals varying from five days to three weeks. Urea in appropriate amount, experimentally determined for each gum, added to a second set regulated the rate of change of viscosity and gelation time to about the same for all. (See last experiment described in this paper.)

Our study of the effect of urea on the gelation of bichromated gum arabic began after we had changed our source of gum for deep-etch coatings. Coatings made from the earlier source had a long shelf-life; those from the later source had a much shorter shelf-life.

Since urea is used in certain papermaking operations successfully as a plasticizer, and has a marked influence on the viscosity and gelation of certain paper coatings, many of carbohydrate nature (*1*), it seemed promising to investigate the effect of urea on bichromated gum arabic solutions.

Consequently many experiments were run on gum from different suppliers at different concentrations, pH and initial viscosities, with and without urea. In all cases, the urea retarded the rate of viscosity increase and time-to-gel. In this paper we are presenting a few interesting illustrations from these experiments.

## EXPERIMENTAL

### *Procedure*

All experiments were performed at room temperature in an air conditioned building over a period of about $1^1/_2$ years. Some experiments took a long time, some were accelerated by choosing appropriate concentrations of materials. Usually the ratio of bichromate-to-gum was of the order of magnitude used in light-sensitive coatings. Most experiments used as a base material 300 ml. of 14°Bé. gum arabic plus 80 ml. of 25% ammonium bichromate solution, to which other chemicals were added either dry or in solution. Where advisable, the bichromate was added last.

Most volumes were within 450 ± 70 ml., which furnished sufficient material for various manipulations. Densities were recorded as °Bé., viscosities in seconds for a Zahn $G_2$ viscosity cup to empty, pH as indicated on an electrometer using the glass electrode. Viscosities were often taken at daily intervals and evidence of gelation was noted.

Table I

| *Sample* → | *A* | *B* | *C* | *D* | *E* |
|---|---|---|---|---|---|
| Gum arabic, g. | 84 | 84 | 84 | 84 | 84 |
| Ammonium bichromate, g. | 17 | 17 | 17 | 17 | 17 |
| Concentrated ammonia, ml. | 13 | 13 | 13 | 13 | 13 |
| Urea, g. | 0 | 0 | 82 | 0 | 0 |
| Magnesium nitrate, g. | 0 | 0 | 0 | 50 | 0 |
| Ammonium nitrate, g. | 0 | 0 | 0 | 0 | 50 |
| Water to make, ml. | 370 | 535 | 535 | 535 | 535 |
| Density, °Bé. | 15.4 | 11.4 | 15.8 | 15.7 | 15.5 |
| pH 8 | 8.8 | 8.7 | 8.8 | 8.5 | 8.3 |
| Viscosity (Zahn $G_2$), sec. | 19.0 | 16.2 | 16.6 | 16.4 | 16.2 |

### *Viscosity Changes with Time on Modified and Unmodified Bichromated Gum Arabic*

Figure 1 shows five curves representing the increase in viscosity with time in bichromated gum arabic solutions ammoniated to pH 8.6, prepared as follows: The ammoniated bichromated gum was divided into five equal portions. Portion *A* was unaltered. Urea was added to *C*, magnesium nitrate to *D*, and ammonium nitrate to *E*. Water was added to *B*, *C*, *D*, and *E* to make them equal in volume, the respective chemicals added to *C*, *D*, and *E* in amount to make their densities approximately the same and equal to that of *A*, *B* was diluted for volume control without regard to density.

Conditions at start of the experiment are tabulated in Table I.

The pH is above the optimum for rapid gelation and the ratio of bichromate-to-gum is less than the optimum, and except for sample *A* the high dilution contributes to delayed gelation. This experiment was designed to run a long time.

Figure 1 illustrates viscosity curves over a period of 16 months. Note that the urea curve, *C*, is practically a straight line and that the viscosity has changed very little over the whole period. The undiluted gum *A* gelled in a little over two months; whereas its diluted counterpart, *B*, did not begin to thicken rapidly until about ten months, nor did it begin to get until considerably over a year.

Both curves *B* and *E* demonstrate rapid change in

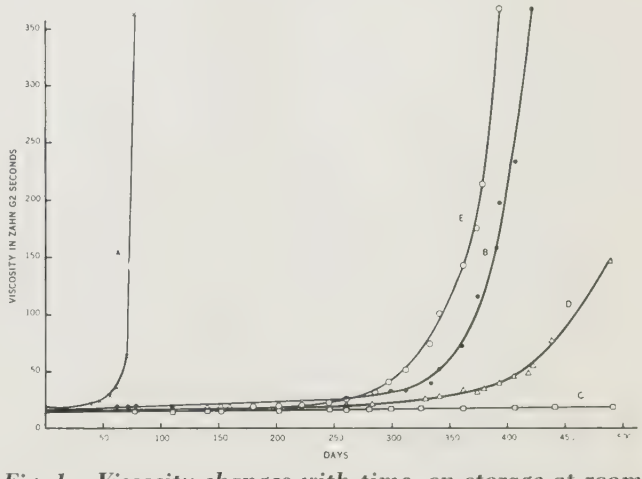


***Fig. 1. Viscosity changes with time, on storage at room temperature, of bichromated and ammoniated gum arabic solutions unmodified, and modified by the addition of certain chemicals and water***

Curve *A*, undiluted bichromated and ammoniated gum 370 ml. volume; Curve *B*, diluted with water to 535 ml.; Curve *C*, urea added and volume made to 535 ml.; Curve *D*, magnesium nitrate added and volume made to 535 ml.; Curve *E*, ammonium nitrate added and volume made to 535 ml. (Note: Composition of these preparations is given in Table I.)

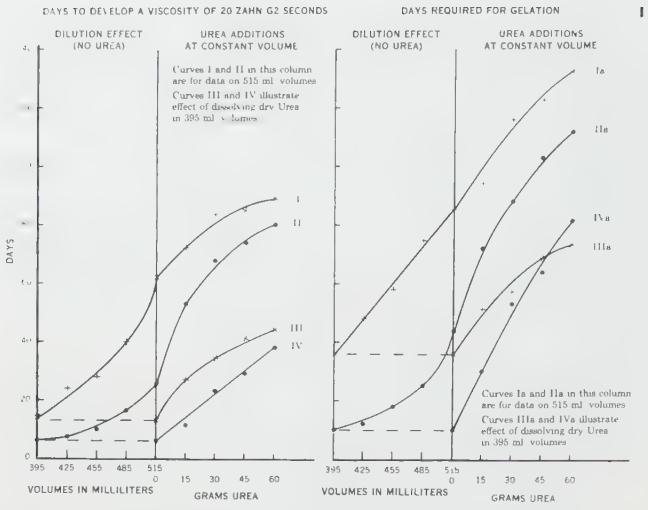
DAYS TO DEVELOP A VISCOSITY OF 20 ZAHN G2 SECONDS
DAYS REQUIRED FOR GELATION
DILUTION EFFECT (NO UREA)
UREA ADDITIONS AT CONSTANT VOLUME
Curves I and II in this column are for data on 515 ml volumes
Curves III and IV illustrate effect of dissolving dry Urea in 395 ml volumes
DAYS
VOLUMES IN MILLILITERS
GRAMS UREA
Curves Ia and IIa in this column are for data on 515 ml volumes
Curves IIIa and IVa illustrate effect of dissolving dry Urea in 395 ml volumes

*Fig. 2. Viscosity and gelation of bichromated gum arabic solutions as affected by concentration, ammoniation, and addition of different amounts of urea*

× Ammoniated and bichromated gum arabic solutions. ● Bichromated gum arabic solutions.

viscosity beginning about the ninth month, but the rate of ammonium nitrate treated gum *E* increased more rapidly. Gelation occurred in about 13 months in *E* and a month later in *B*. Both the gels were very soft and lumpy. Preparation *D* with magnesium nitrate became very thick, but did not gel until after 16 months.

During this whole period, portion *C* (urea treated) did not exceed 20 Zahn $G_2$ seconds viscosity. It is clearly demonstrated in this experiment that urea had a definite retarding influence on the gelation of ammoniated bichromated gum arabic and on the rate of increase in viscosity.

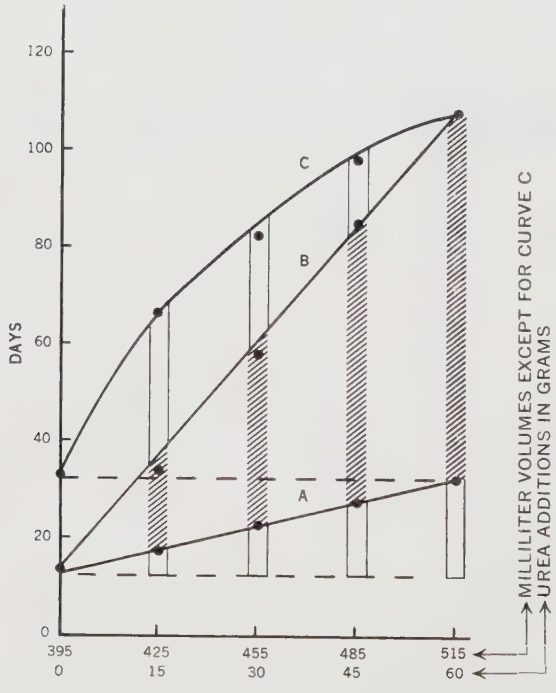


*Fig. 3 Effect of dilution, and of urea additions on gelation time of a bichromated gum arabic solution at 4.3 pH. (380 ml. bichromated gum was made to the volumes indicated)*

Curve *A*, days-to-gel as influenced by dilution only; Curve *B*, days-to-gel when urea is added with the water, in the amounts shown; Curve *C*, days-to-gel when all volumes are made to 515 ml.; Curves *A*, *B* and *C* illustrate three sets of experiments run parallel.

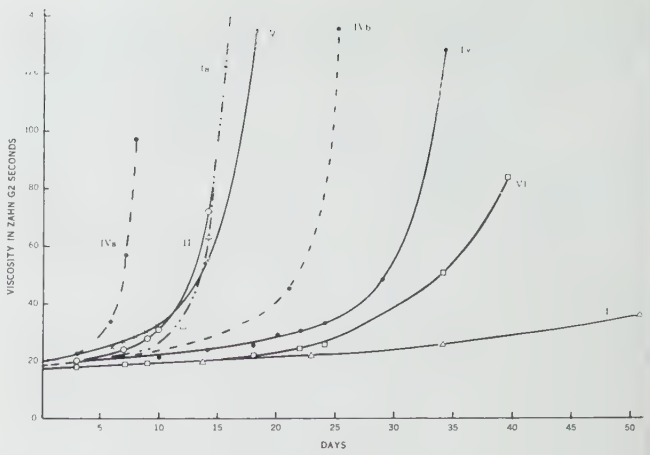


*Fig. 4. Viscosity curves of bichromated commercial gum arabic solutions, 14° Bé. and 4.3 pH, with and without urea additions*

*I*, 30 g. of urea added to 380 ml. bichromated gum; *Ia*, no urea added; *II*, 30 g. of urea added; *IV*, 30 g. of urea added; *IVa*, no urea added; *IVb*, 15 g. of urea added; *V*, 30 g. of urea added; *VI*, 30 g. of urea added. Roman numerals refer to source of gum.

### *Viscosity and Gelation of Bichromated Gum Arabic Solutions as Affected by Concentration, Ammoniation, and Addition of Different Amounts of Urea*

Figure 2 illustrates the comparison of the effect of dilution, ammoniation, and urea additions to bichromated gum arabic solutions, in respect to time required to reach a viscosity of 20 Zahn $G_2$ seconds, and to gel. All preparations were made with the same amount and kind of gum containing the same amounts of ammonium bichromate, i.e., 300 ml. of 14°Bé. gum and 80 ml. of a 25% solution of bichromate.

Preparations represented by curves *I*, *Ia*, *III*, *IIIa* are ammoniated with 15 ml. concentrated ammonia to pH 9. Preparations represented by *II*, *IIa*, *IV*, *IVa* are not ammoniated.

The left portions of curves *I* and *II* lying in the column headed Dilution Effect illustrate the time in days in which a viscosity of 20 Zahn $G_2$ seconds was developed by preparations made to the volumes stated at the bottom of the chart, 395, 425, 455, 485, 515 ml. Increasing dilution, of course, delays the rate of vis-

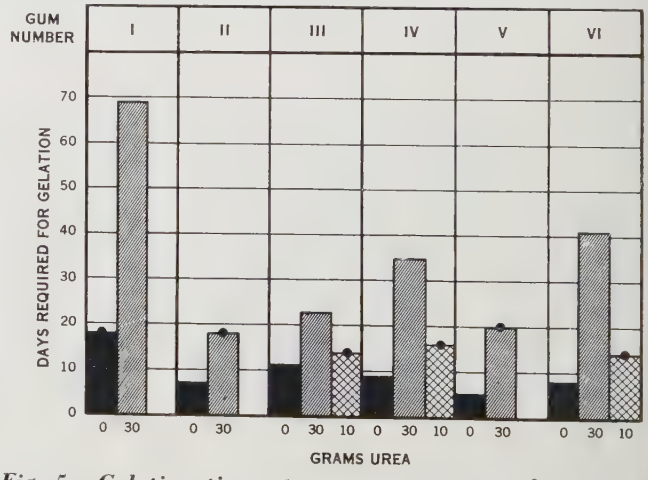


*Fig. 5. Gelation time at room temperature for unammoniated bichromated gum arabic solutions prepared from different commercial sources of stabilized gum, with and without urea additions*

● Gelled in 17 ± 3 days. ■ No urea per 380 ml. bichromated gum. ▨ 30 g. urea per 380 ml. bichromated gum. ▩ 10 g. urea per 380 ml. bichromated gum.

cosity increase in both curves, the alkaline solutions being delayed the longer.

Continuing these curves (*I* and *II*) into the column headed Urea Additions at Constant Volume illustrates the effect on viscosity development when 15, 30, 45, and 60 g. of urea are added to solutions of a constant, 515-ml. volume. Increases in urea extend the delay of viscosity development in both the ammoniated and unammoniated bichromated solutions.

The next two columns of the chart (curves *Ia, IIa, IIIa, IVa*) portray similar data applying to gelation of the ammoniated and unammoniated bichromated gum arabic. Dilution with water and addition of urea as in the viscosity testing, demonstrate corresponding delays in gelation.

Urea is thus shown to retard increase in viscosity as well as gelation time in both ammoniated and unammoniated bichromated gum arabic solutions.

#### *Relative Effect of Dilution and Urea Additions on the Gelation Time of a Good Commercial Gum Solution at a pH of 4.3*

Figure 3 illustrates another experiment with a different gum arabic solution. The unammoniated data are plotted to show the relative effect of dilution and urea additions.

Curve *A* shows the delay in gelation due to dilution with water. Curve *B* represents the combined effect of adding urea along with the water. Curve *C* shows the effect of bringing all the solutions to a volume of 515 ml. before adding urea. The open columns show the effect of dilution only. The shaded columns show the effect of the urea. Curves *A* and *B* turned out to be straight lines in this experiment.

Undiluted, the bichromated gum gelled in 13 days. Diluting to 515 ml. delayed the gelation to 32 days. The 32 days were extended to 107 days when 60 g. of urea were included, which is another example of the retarding of gelation by urea.

It is to be noted that these preparations are acidic. If ammoniated to approximately pH 9, the time would be considered extended.

#### *Comparison of Viscosity and Gelation Characteristics of Several Different Commercial Gum Arabic Solutions Supplied at 14°Bé. and pH 4.3 ± 0.1*

Gum arabic solutions from several commercial sources were compared as to viscosity changes and gelation time addition of ammonium bichromate.

A volume of 80 ml. of 25% bichromate solution were added to 300 ml. of each gum at 14°Bé. and pH 4.3. Viscosity curves were plotted and gelation time noted.

Dry urea (30 g.) were dissolved in corresponding amounts of the same gums and bichromated in the same way. Viscosity curves were plotted and gelation noted as for the samples without urea. Smaller amounts of urea were added to some samples and related changes were observed. Urea had the usual delaying effect on viscosity and gelation, but to a different extent on gums from different sources.

In Fig. 4, several viscosity curves are shown. The solid lines are curves of solutions to which 30 g. urea were added. The broken lines illustrate differences due to smaller addition and no addition of urea. Roman numerals indicate source of gum, *a* indicates samples containing no urea, *b* indicates addition of 15 g. of urea.

Figure 5 shows the result of a rough attempt to bring all the bichromated gums of the several commercial sources to approximately the same gelation time by adding appropriate amounts of urea. The height of the bars represents the time in days for the variously treated samples to gel. The bars topped with a black disk indicate the treatments resulting in the closest agreement with a constant gelation time for the series.

The gums are not identical in their gelation responses to the addition of ammonium bichromate alone, or to the further addition of equal amounts of urea. In the six cases illustrated, urea requirement to hold gelation time to 17 ± 3 days varied from 0 to 30 g. per 380 ml. bichromated gum. Nevertheless, appreciable amounts of urea always extended the gelation time.

In line with the testing for gelation in the above gums, it is conceivable that different gums exhibiting various gelation levels when bichromated, might be blended in such proportions that a mixture possessing satisfactory properties would result.

## DISCUSSION

In 1937 Haller and Frankfurt (*2*) published the results of studies of the effects of the chlorides, sulfates and nitrates of chromium, aluminum and iron on purified gum arabic solutions. These trivalent metallic salts added to gum arabic affect the viscosity of the gum and coagulate it. The gels formed have the colors of the oxides of the corresponding metals. The oxides themselves mixed with the gum do not cause corresponding gelation, so the gelling is attributed to so-called "nascent" oxide which forms an absorption complex with the gum.

By dialyzing a metallic chloride-gum mixture, HCl is obtained, demonstrating that the cation is responsible for the gel.

The function of urea in delaying the effect of the chromium appears to be that of a stabilizer, possibly acting in some way as an antioxidant. Natural gum arabic contains both oxidases and perioxidases.

#### *Prediction of Relative Gelation Time*

If to the freshly bichromated gum an appropriate volume of 50% chromic acid solution is added, gelation will occur in minutes. In less than 8 hr. the order in which the gums of a series will gel may be predicted. For example, in a series of three different freshly bichromated gums to which 10% by volume of 50% chromic acid solution was added, gelation occurred in 190, 230, and 300 minutes, respectively. The corresponding bichromated gums to which no chromic acid was added gelled in 6, 11, and 24 days, respectively. Precise, rapid, and identical technique is essential (one sample at a time) in applying this test.

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# Examination of Western Hemlock for Lignin Precursors

OTTO GOLDSCHMID and H. L. HERGERT

**Cambium and sapwood of western hemlock were examined for the presence of low-molecular-weight components which might be intermediates in lignin formation. It was believed that this knowledge would provide further information on the structure of lignin. The following extractive substances were detected in the cambium: quinic acid, shikimic acid, sucrose, fructose, arabogalactan, coniferin, leucocyanidin (3′,4′,5,7-tetrahydroxyflavan-3,4-diol), catechin, epicatechin, and depsides of the following acids: caffeic-shikimic, ferulic-shikimic, ferulic-quinic, and *p*-coumaric-shikimic. A fifth depside was tentatively identified as *p*-coumaryl-quinic acid. In addition to these compounds, several glycosides of lignanlike compounds were detected, but their structure has not yet been completely elucidated. The following compounds were isolated from, or detected in, the sapwood: Brauns "native lignin," conidendrin, hydroxymatairesinol, oxomatairesinol, pinoresinol, dehydrodiconiferyl alcohol, vanillic acid, vanillin, coniferylaldehyde, ferulic acid, fumaric acid, sequoyitol, pinitol, quinic acid, shikimic acid, catechin, leucocyanidin, xylose, arabinose, mannose, glucose, galactose, rhamnose, and several incompletely characterized lignan glycosides. The presence of guaiacylglycerol-β-coniferyl ether was indicated but not definitely ascertained. Depsides were absent. The presence in the cambium of phenolic glucosides and the absence of Freudenberg's proposed intermediates in lignin formation indicate that either these intermediates are of such a transitory nature that they cannot be detected, or that lignin formation proceeds *via* glucoside intermediates. Not directly related, but of interest, was the observation that conidendrin is formed during a sulfite cook by dehydration of hydroxymatairesinol.**

Some of the intermediates in lignin biosynthesis have been determined with reasonable certainty, e.g., glucose, shikimic acid, phenylpyruvic acid, and coniferin. However, the intermediates that would actually provide the most information on the structure of lignin are those between coniferin and lignin, and these have not yet been established unequivocally. Speculation concerning the nature of these compounds is based largely on the *in vitro* biosynthetic experiments of Freudenberg (*1*).

In 1949 Freudenberg began an investigation of the formation of ligninlike dehydrogenation polymers resulting from the enzymatic dehydrogenation of coniferyl alcohol. The origin of Freudenberg's investigation may be traced back to Klason's hypothesis that lignin is derived from coniferyl alcohol and Erdtman's idea that lignin is formed by enzymatic dehydrogenation of a guaiacyl-propane derivative. Since then Freudenberg and his collaborators have been able to isolate and identify a large number of intermediates in, and to clarify greatly the mechanism of, the enzymatic dehydrogenation of coniferyl alcohol (I) and other hydroxy cinnamyl derivatives. (Fig. 1.)

The intermediates that were first identified, and that were present in largest amounts, were the following dimers: dehydrodiconiferyl alcohol (II), DL-pinoresinol (III), and guaiacylglycerol-β-coniferyl ether (IV). Freudenberg called these dimers secondary lignin building units because they in turn form ligninlike products by enzymatic dehydrogenation polymerization. The enzyme used in all these experiments was a phenoloxidase obtained from the mushroom *Psalliota campestris*. More recently this enzyme has been characterized as a laccase (*2*). Similar results were obtained with peroxidase in the presence of hydrogen peroxide, with copper sulfate and oxygen, and, though very slowly, even in the presence of air and water without any other additions. Furthermore, it was found that an enzyme preparation obtained from spruce cambium also catalyzed the dehydrogenation polymerization of coniferyl alcohol.

In 1953 Freudenberg and Dietrich reported (*3*) that they had found small amounts of coniferyl alcohol and of the three secondary building units in fresh cambial sap of spruce by paper chromatography and color reactions, but no experimental details were provided. In 1956, however, Freudenberg mentioned (*4*) that attempts to identify these dimers in cambial sap had not lead to clear results, and that small amounts of coniferyl alcohol and of II and III could have formed during sample preparation. In 1958 Freudenberg and Niedercorn stated (*5*) that cambial sap, obtained by first inactivating the enzymes with formaldehyde solution, contained coniferin, sugars, small amounts of coniferyl alcohol and quinic acid, and unknown substances of higher molecular weight that gave a yellow or orange-yellow color reaction with diazotized sulfanilic acid. It was also stated that hydroxymatairesinol and pinoresinol were present, possibly belonging to the resin extractives. On standing, cambial sap obtained without the formaldehyde treatment contained, in addition to the previously mentioned compounds, shikimic acid, protocatechuic acid, the three dimers, II, III, and IV, and other intermediates. Again, no experimental evidence was cited.

Our work began in 1955 with the following hypothesis: if II, III, and IV were intermediates in the formation of lignin, these compounds should be present in the cambium or in the zone adjacent to the cambium of a tree where lignin is being synthesized. An examination of cambial constituents was therefore initiated in an attempt to find such intermediates. If these or other compounds could be found, it was believed this knowledge would provide further information concerning the structure of lignin. Western hemlock was selected as the species for study, since a large number of hemlock trees of varying age were available in the immediate vicinity of our laboratory. Furthermore, the wood of this species does not contain resin canals and is relatively low in resinous constituents. It was hoped that this would simplify the isolation of phenols present in very low concentration.

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## EXPERIMENTAL

Ultraviolet spectra were determined on a Cary Model 11 recording spectrophotometer. Infrared spectra were obtained as paraffin oil (Nujol) mulls, films deposited from an appropriate solvent, or as a potassium bromide wafer on a Perkin-Elmer Model 21 double-beam spectrophotometer. Elemental analyses were conducted by Weiler and Strauss, Oxford, England. All melting points are corrected unless otherwise stated.

### *Paper Chromatography*

Paper chromatography was conducted on Whatman no. 1 paper by the descending technique one-dimensionally in glass jars or two-dimensionally in a Reco A-300 Chromatocab tank. The following developers were used:

*a.* Butanol-acetic acid-water (4:1:5, organic phase) (*6*).
*b.* Toluene–acetic acid–water (4:1:5, organic phase).
*c.* Benzene–ligroin–methanol–water (50:50:1:50, organic phase) (*7*).
*d.* Methyl ethyl ketone saturated with water (*8*).
*e.* *n*-Butanol saturated with water.
*f.* Acetone–water (95:5) (*9*).
*g.* Ethyl acetate–pyridine–water (8:2:1, organic phase).
*h.* Water saturated with benzene (*10*).
*i.* Two per cent acetic acid (*11*).
*j.* Carbon tetrachloride–dimethylformamide (9:2 or 11:3, Whatman no. 2 or no. 11 paper) (*5*).

Solvent systems *b*, *c*, and *d* could only be used when the atmosphere of the tank was thoroughly saturated. This was most conveniently accomplished by irrigating sheets in adjacent troughs with the aqueous solvent phase.

After development, sheets were thoroughly air-dried and then examined under an ultraviolet lamp before and after fuming with ammonia vapor. They were then sprayed with different reagents to distinguish various compounds. Spray reagents used were:

*a.* Ferric chloride-potassium ferricyanide (*12*).
*b.* Diazotized sulfanilic acid (*13*).
*c.* Bis-diazotized dianisidine (*7*).
*d.* Cinnamaldehyde-hydrochloric acid (*28*).
*e.* Phloroglucinol-hydrochloric acid (*28*).
*f.* Permanganate-periodate (*14*).
*g.* *p*-Anisidine-trichloro acetic acid.
*h.* Periodate-nitroprusside-piperazine (*15*).
*i.* Naphthoresorcinal-trichloroacetic acid (*6*).

The concentrations used were those described in the literature.

In order to detect compounds containing an alpha-hydroxyguaiacyl and/or a guaiacylglycerol nucleus, two special techniques were used.

The test for an alpha-hydroxyguaiacyl nucleus was based on Ziegler's observation (*16*) that *p*-hydroxyphenyl derivatives with a carboxyl or an alpha-hydroxyl group are cleaved during coupling by a diazo reagent. In our laboratory it was observed that the same compound is formed by reaction of guaiacol, vanillic acid, vanillyl alcohol, or apocynol [1-hydroxy, 1-(3-methoxy, 4-hydroxyphenyl) ethane] with diazotized sulfanilic acid. To conduct the test, the compound suspected to contain an alpha-hydroxyguaiacyl nucleus is spotted on Whatman no. 1 paper. Adjacent to the compound, a spot of guaiacol or vanillic acid is spotted. A spot of a butanol solution of diazotized sulfanilic acid (same concentration as spray reagent *b*) is placed on top of the spotted compounds and allowed to dry

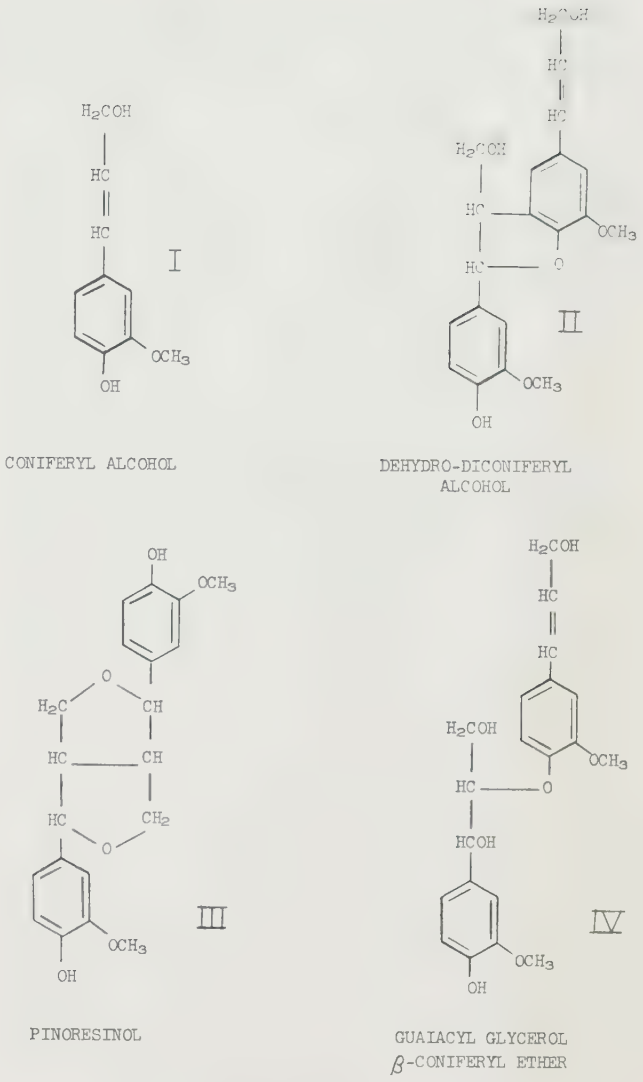



*Fig. 1*

for 5 min. A spot of 10% sodium carbonate solution is then placed on the coupled phenol, dried, and the chromatogram developed with solvent *d* for 10 to 24 hr. (*8*). A positive test consists of the formation of a bright orange dye with the same $R_f$ as that derived from guaiarol or vanillic acid. Alternately, the paper spotted with the compounds may be lightly sprayed with the diazotized sulfanilic acid and sodium carbonate solutions, dried, and then developed.

The colors obtained by coupling of various phenols with diazotized sulfanilic acid (spray reagent *b*) were diagnostic. Lemon-yellow colors were obtained with para hydroxyphenyl derivatives, bright orange with alpha-hydroxyguaiacyl derivatives, guaiacol, or vanillic acid, pink colors with guaiacyl derivatives with a $CH_2$ or branched chain in the alpha position, e.g., conidendrin, and bright magenta with a guaiacyl compound with alpha-beta unsaturation, e.g., coniferyl alcohol or ferulic acid.

Detection of a guaiacylglycerol nucleus was conducted by dehydration to form coniferylaldehyde. The compound suspected to contain the guaiacylglycerol nucleus was dissolved or suspended in dioxane-concentrated hydrochloric acid (99:1) and refluxed for 1 to 2 hr. The mixture was concentrated and spotted

**Table I. Composition of Cambial Material**

| Percentages based on O.D. weight of original material | |
|---|---|
| Ethanol soluble | 56.2 |
| Cold-water soluble | 12.4 |
| Hot-water soluble | 4.4 |
| Extracted residue | 27.0 |

**Table II. Composition of the Carbohydrate Fraction of the Insoluble Cambial Residue**

| Percentages based on total carbohydrate fraction | |
|---|---|
| Glucose | 58.8 |
| Mannose | 13.4 |
| Galactose | 12.8 |
| Arabinose | 9.1 |
| Xylose | 5.9 |

directly or taken up in benzene, filtered, and then spotted on Whatman no. 1 paper. The chromatograms were developed with solvent systems *c* and *i*, dried, and then sprayed with reagents *b* and *e*. The detection of coniferylaldehyde, alpha-hydroxypropiovanillone and acetyl vanilloyl was considered to be a positive test. Further details concerning this test will be described elsewhere.

***Cambium***

*Collection and Preparation.* Samples of cambial sap and tissue were obtained from Western hemlock (*Tsuga heterophylla*) trees growing near Shelton, Wash., in September of 1955 and in May, June, and July, 1956. During the active growing period of the tree, the bark can be peeled off readily. The freshly exposed wood surface was scraped with a spatula, and the cambial sap and scrapings collected in a container filled with ethanol, methanol, or methyl ethyl ketone. The samples were allowed to stand in the solvent overnight. After this period, the extracted solids were separated from the solution by centrifuging or filtration.

*Summative Analysis.* A typical composition of a sample of cambial material (September, 1955) that was exhaustively extracted in succession with ethanol, cold water, and hot water, is shown in Table I. The extracted residue was analyzed for nitrogen (4.23%), methoxyl (2.6%), and ash (0.75%). The monomeric sugar constituents of the residue were determined after total hydrolysis by the paper chromatographic method of Jeffery, Partlow, and Polglase (*17*). The composition of the carbohydrate fraction (18.7% of the residue) is shown in Table II.

*Coniferin.* A sample of cambial material (May, 1956) was extracted with methanol at room temperature. The filtered extract was concentrated at room temperature in a circulating evaporator. The concentrated solution which now was aqueous was extracted with ether. Upon further concentration of the ether-extracted solution, a crystalline precipitate formed which was filtered off. The crystals were washed with a small amount of water, which was combined with the filtrate. The combined filtrates were concentrated and set in a refrigerator, where more crystals formed.

The combined crystalline material (14.8% of the alcohol-soluble solids) was recrystallized from hot water to give white needles of coniferin, m.p. 183–184°C., $[\alpha]_D^{25}$ −66.7 (*c*, 0.5 in water) [lit. (*18*) for coniferin, m.p. 185°C. $[\alpha]_D^{20}$ −66.9]. The acetate derivate was prepared by treatment with acetic acid and pyridine. White needles, softening point 90°C., m.p. 130°C. (lit. (*18*) softening 90°C., m.p. 128–129°C.) were obtained after recrystallization from 80% ethanol. The UV absorption spectrum of coniferin (Fig. 2) showed maxima at 214, 258, and 294 mμ with log ε 4.42, 4.28, 3.78, respectively.

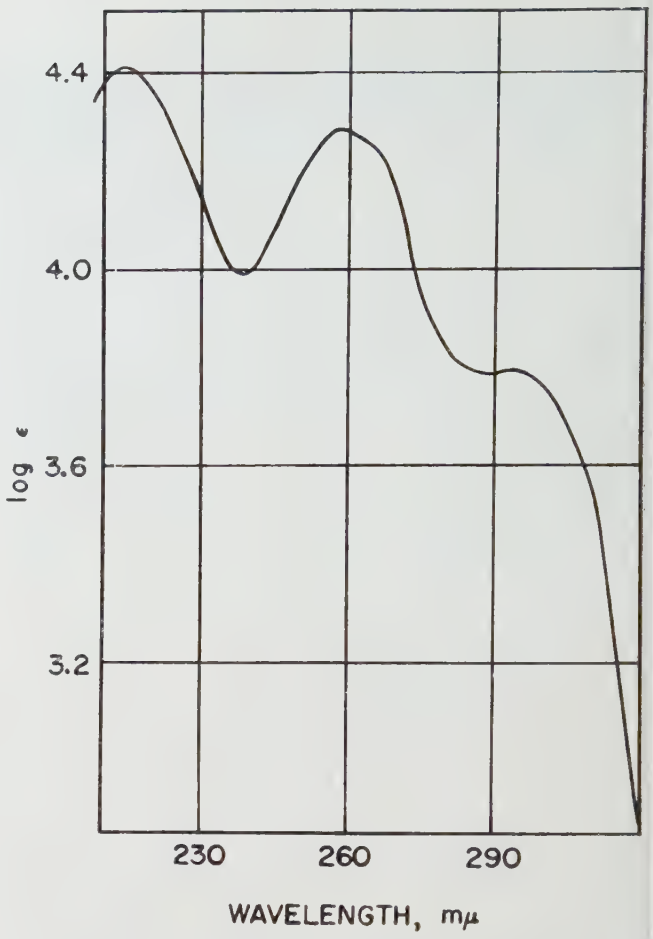



***Fig. 2. Ultraviolet absorption spectrum of coniferin in water ($6.6 \times 10^{-5}M$)***

Coniferin (50 mg.) in 18 ml. water was mixed with 20 ml. of clear filtrate from a 1% suspension of emulsin (Bios Laboratories, Inc.) and enough buffer to give 50 ml. of a solution at pH 4.4 (acetic acid–sodium acetate, 0.2*N*) and allowed to stand 20 hr. at 30°C. The hydrolyzed solution was neutralized with bicarbonate solution, concentrated, and thoroughly extracted with ether. The ether extract was carefully evaporated to dryness under nitrogen. The residue was dissolved in 5.0 ml. of ethanol and stored in a refrigerator. Ultraviolet absorption spectra of solutions in ethanol and 0.06*N* NaOH solution showed $\lambda_{max.}$ values of 263 and 287 mμ, respectively [lit. (*19*) coniferyl alcohol, $\lambda_{max.}$ 261 and 287 in ethanol and *N* NaOH, respectively]. A two-dimensional paper chromatogram (solvents *a* and *h*) showed one spot only with $R_f$ values 0.82 and 0.58, respectively, after spraying with *b*. When this solution, which was used as a chromatographic standard for coniferyl alcohol, was rechromatographed two years later in solvent *j*, the chromatograms showed in addition to coniferyl alcohol, small spots that agreed in $R_f$ with pinoresinol and dehydrodiconiferyl alcohol.

*Paper Chromatography of Cambial Extracts.* The

water-soluble and the ether-soluble fractions of the alcoholic cambial extracts were investigated by one- and two-dimensional chromatography with a variety of solvents and spray reagents. These studies revealed the presence in the extracts of a large number of individual compounds. A typical chromatogram is shown in Fig. 3, which illustrates the appearance of phenolic compounds after separation with solvents *a* and *i* and development with spray *b*. This group includes catechinlike compounds, depsides of cinnamic acid derivatives, and other phenols. Other chromatograms indicated the presence of soluble carbohydrates, and of quinic and shikimic acids. Most of these compounds were present in the cambial extracts in small amounts which precluded isolation; they were identified by ultraviolet spectrophotometry, chromatographic $R_f$, color reactions, and comparison with authentic compounds as described below:

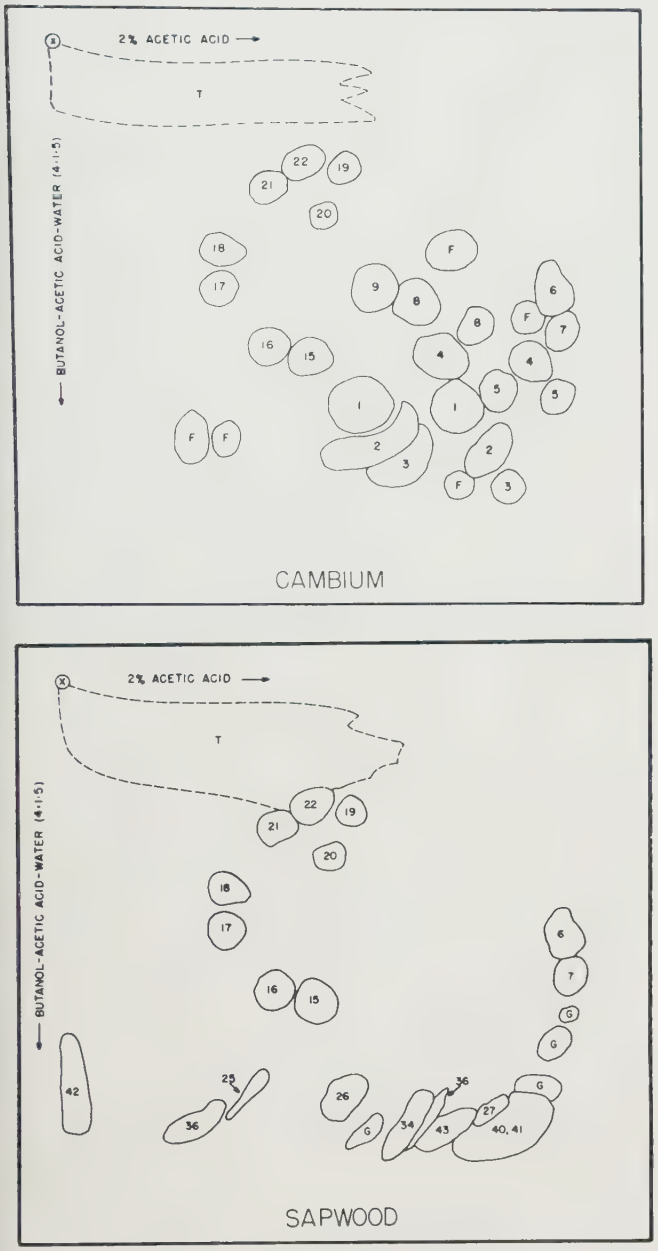



***Fig. 3. Two-dimensional chromatograms of the methanol extracts of western hemlock cambium and sapwood. Identified spots are numbered as follows:***

1. Caffeyl-shikimic acid.
2. Ferulyl-shikimic acid.
3. *p*-Coumaryl-shikimic acid
4. Ferulyl-quinic acid.
5. *p*-Coumaryl quinic acid.
6 and 7. Phenolic glucosides.
8 and 9. Phloroglucinol derivatives.
15. Catechin
16. Epicatechin
17. Gallocatechin.
18. Epigallocatechin.
19–22. Leucocyanidins.
25. Coniferyaldehyde.
26. Vanillin.
27. Vanillic acid.
34. Pinoresinol.
36. Ferulic acid.
40. Hydroxymatairesinol.
41. Oxomatairesinol.
42. Conidendrin.
43. Matairesinol.
F. Unidentified substances fluorescent under ultraviolet light.
G. Unidentified guaiacyl derivatives.
T. Polymeric tannins.

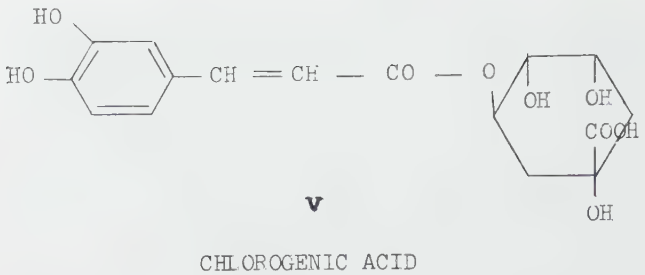
*Soluble Carbohydrates.* The filtrate from the crystallization of coniferin, chromatographed with solvents *a* or *g* and developed with sprays *g* and *i* showed spots corresponding in $R_f$ and color reaction with those of authentic samples of sucrose, fructose, glucose, and galactose. The amounts of sucrose and fructose were estimated by visual comparison with standards to be in the order of 15 to 20%, and 5 to 10%, respectively, based on the O.D. weight of the alcohol soluble solids.

Quantitative paper chromatographic analysis (*17*), after hydrolysis, of the combined solids from the cold and hot water extracts after alcohol extraction of the cambial material (see Table I) gave 5.4% galactose, 4.7% arabinose, and a trace of xylose (based on the O.D. weight of the total water-soluble solids).

*Quinic and Shikimic Acids.* A sample of cambial material (June, 1956) was extracted with methyl ethyl ketone. Upon concentrating, the extract split into an aqueous and an organic phase. The aqueous phase was concentrated to a small volume, and chromatographed two-dimensionally with solvents *a* and *i*. Spray *h* revealed two greenish yellow spots corresponding with authentic quinic acid and shikimic acid, $R_f$ 0.20 and 0.40 in solvent *a*, $R_f$ 0.90 and 0.85 in solvent *i*, respectively. The color reaction with spray *h* is given only by quinic and shikimic acids and a few related compounds such as dehydroshikimic acid (*20*), whose $R_f$ values in solvent *a* and shade of color differ sufficiently from those of quinic and shikimic acids to make their positive identification possible. The spray was used as described by Cartwright and Roberts (*15*), except for omitting the final heating of the chromatogram. When the heating is omitted, the spots appear more slowly but at sufficiently different rates to provide an additional distinction between quinic acid and shikimic acid. The yellow spot due to shikimic acid appears before that from quinic acid.

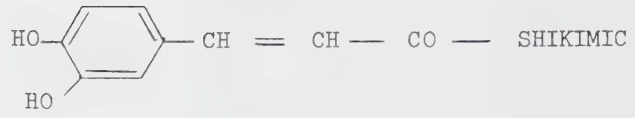
*Depsides with Quinic and Shikimic Acid.* Two-dimensional chromatograms with solvents *a* and *i* of the alcoholic extracts of cambial material from Western hemlock showed a number of spots with a strong fluorescence under ultraviolet illumination. The color of the fluorescence, the change in this color on fuming with ammonia and the pair-wise arrangement of some of these spots were similar to those of chlorogenic acid (V), an ester (or depside) of caffeic acid with quinic acid (Fig. 4).

In order to isolate these compounds, aliquots of the concentrated aqueous phase of a methyl ethyl ketone (MEK) extract of cambial material were spotted on Whatman no. 3 mm. paper. Two-dimensional chromatograms were prepared with solvents *a* and *i*. The areas corresponding to the fluorescent compounds 1 to 5, marked under ultraviolet illumination, were cut out and eluted with methanol. Ultraviolet absorption spectra of the eluates were determined in

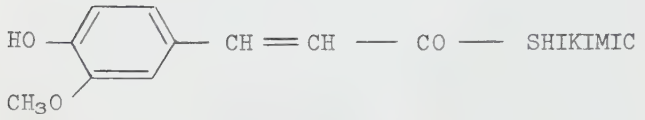



V

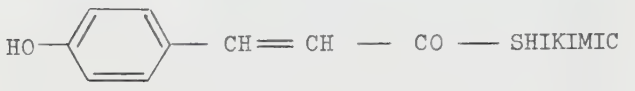
CHLOROGENIC ACID



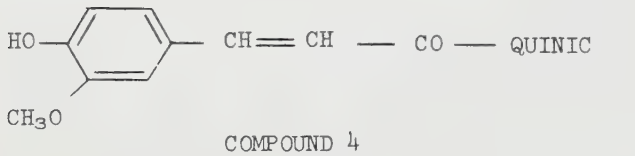

COMPOUND 1




COMPOUND 2




COMPOUND 3




COMPOUND 4

*Fig. 4*

neutral and alkaline solutions. The spectrum of compound 1 was similar to that of chlorogenic acid. The spectra of the eluates of compounds 2 and 3 were similar to those of the methyl esters of ferulic and *p*-coumaric acid, respectively. This indicated that the compounds were probably depsides of cinnamic acid derivatives which are known (*21*) to give rise to pairs of spots when chromatographed with solvent *i*. Their $R_f$ values, however, did not correspond with those of any of the known isomers of chlorogenic acid.

In order to identify the components of the depsides, compounds 1 to 4 (Fig. 4) were hydrolyzed as follows: The eluates were evaporated to dryness, and the residues treated for 4 hr. on a steam bath with small amounts of 2% HCl. Acid hydrolysis gave results superior to those of the commonly used alkaline hydrolysis, since quinic and shikimic acid could be more readily recovered.

The hydrolyzates were thoroughly extracted with ether, the ether extracts and the extracted hydrolyzates concentrated to a small volume, dried in a vacuum desiccator, and taken up in acetone. The ether-soluble components of the hydrolyzates of compounds 1 to 3 chromatographed with solvent *a* and sprayed with *b* corresponded in $R_f$ value and color reaction with known samples of caffeic acid, ferulic acid, and *p*-coumaric acid, respectively; the ether-insoluble components, chromatographed with the same solvent and

**Table III. A Comparison of the Ultraviolet Characteristics of Cambial Eluates with Known Cinnamoyl Derivatives**

| | $\lambda_{max.}$, mμ | |
|---|---|---|
| | *Neutral* | *Alkaline* |
| Compound 1 | 328 | 358 |
| Chlorogenic acid | 324 | 364 |
| Compounds 2 and 4 | 325 | 372 |
| Methyl ferulate | 325 | 367 |
| Compound 3 | 312 | 358 |
| Methyl *p*-coumarate[a] | 308 | 352.5 |

[a] Geissman, T. A. and Harborne, J. B., *Arch. Biochem. Biophys.* 55: 447 (1955).

sprayed with *h* agreed in $R_f$ value and color reaction with authentic shikimic acid. Compounds 1, 2, and 3 were thus shown to be caffeoyl-shikimic, feruloyl-shikimic, and *p*-coumaryl-shikimic acid, respectively. By similar treatment, compound 4 was shown to be feruloyl-quinic acid. Compound 5 could not be eluted in sufficient amount for positive identification of its components, but based on inspection of $R_f$ values and color reaction, it is probably *p*-coumaryl-quinic acid (Table IV).

*Phenolic Glucosides.* In addition to the strongly fluorescent depsides, two-dimensional chromatograms of cambial extracts showed two spots that gave a strong orange-yellow color with spray *b*, a positive reaction with spray *a*, but did not fluoresce under ultraviolet illumination: Compound 6, $R_f$ values in solvent *a* and *i*, 0.45 and 0.80, and compound 7, $R_f$ values in the same solvents, 0.55 and 0.80, respectively. These compounds could not be obtained in sufficient amounts for further investigation from cambial extracts. Since chromatograms of extracts from the sapwood showed the identical spots, and sapwood extracts could be more readily procured, an attempt was made to isolate compounds 6 and 7 from these extracts.

The butanol-soluble portion of fraction B from the sapwood (see below), upon further concentration split into a butanol and an aqueous phase. Both phases were then further fractionated. The butanol-soluble phase was separated into 78 fractions on a filter paper column (Grycksbo) using solvent *e*. Fractions 39 to 54 which contained compound 6 were combined and evaporated to dryness. The water-soluble phase was ap-

**Table IV. $R_f$ Values and Color Reactions of Cambial Compounds 1 to 5**

| *Before hydrolysis* | | $R_f$ | | | *Color* | |
|---|---|---|---|---|---|---|
| *No.* | *Compound* | *Solvent* a | | *Solvent* i | *UV + $NH_3$* | *Spray* b |
| 1 | Caffeoyl-shikimic | 0.67 | 0.47 | 0.64 | Yellow-green | Yellow |
| 2 | Feruloyl-shikimic | 0.74 | 0.50 | 0.66 | Blue-green | Purple |
| 3 | *p*-Coumaryl-shikimic | 0.78 | 0.54 | 0.68 | Purple | Pink |
| 4 | Feruloyl-quinic | 0.58 | 0.59 | 0.74 | Blue-green | Purple |
| 5 | (*p*-Coumaryl-quinic?) | 0.62 | 0.67 | 0.81 | Purple | Pink |

| *Hydrolyzate, ether-soluble* | | $R_f$, *Solvent* a | | *Color* | |
|---|---|---|---|---|---|
| | | *Lit.*[a] | *Found* | *UV + $NH_3$* | *Spray* b |
| From 1 | Caffeic acid | 0.78 | 0.80 | Faint blue | Faint brown |
| From 2 and 4 | Ferulic acid | 0.84 | 0.84 | Bright blue | Purple |
| From 3 | *p*-Coumaric acid | 0.88 | 0.87 | Purple | Pink |

| *Hydrolyzate, ether-insoluble* | | $R_f$ *Solvent* a | *Color Spray* h |
|---|---|---|---|
| From 1, 2, and 3 | Shikimic acid | 0.40 | Greenish yellow |
| From 4 | Quinic acid | 0.20 | Greenish yellow |

[a] Swain, T., *Biochem. J.* 53: 200 (1953).

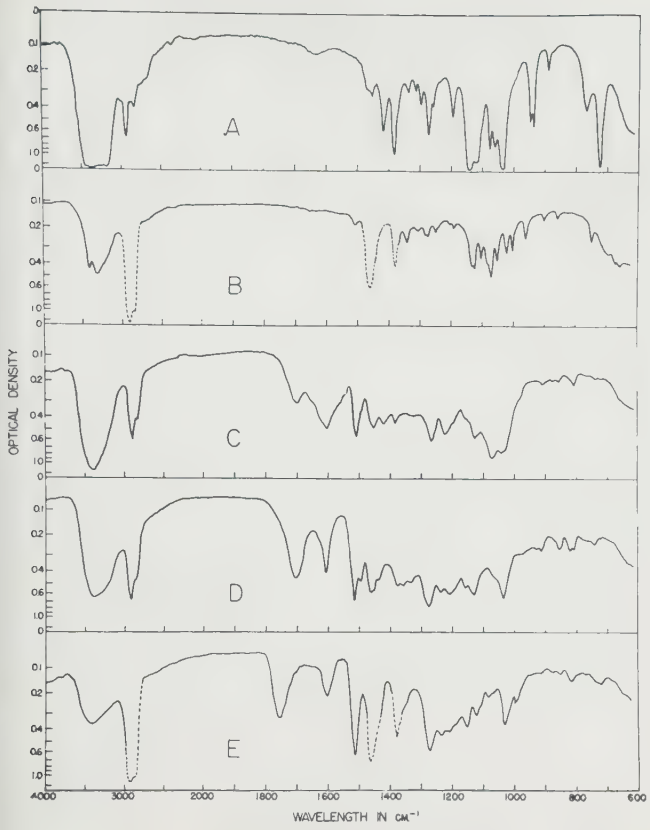



*Fig. 5. Infrared absorption spectra of (a) sequoyitol (potassium bromide wafer), (b) pinitol (paraffin oil mull), (c) phenolic glucoside (compound 6) (potassium bromide wafer), (d) aglycone of compound 6 (film), (e) hydroxymatairesinol (paraffin oil mull)*

plied as streaks on the starting line of several sheets of Whatman no. 17 paper to which had been sewn wicks of Whatman no. 50 in order to decrease the rate of flow of the solvent (*22*). After chromatographic separation with solvent *e*, zones containing compounds 6 and 7, respectively, were eluted with methanol.

The infrared spectrum of compound 6 was measured as a potassium bromide wafer (Fig. 5). Since the infrared spectrum of compound 6 indicated it to be a glucoside, it was hydrolyzed with emulsin exactly as described for coniferin, and also with dilute sulfuric acid. The hydrolyzates were thoroughly extracted with ether. Two-dimensional chromatograms of the ether-soluble component showed one spot with $R_f$ values 0.84 and 0.65 in solvents *a* and *i*, respectively, which gave an orange-pink color reaction with spray *b*. The ether-extracted acid hydrolyzate was neutralized with IR-4B resin, filtered, and concentrated to a small volume. A chromatogram of this solution with solvent *a*, sprayed with spray *g* gave a strong spot corresponding in $R_f$ value and color reaction with glucose.

Ultraviolet absorption spectra (Fig. 6) of compound 6 and of the aglycone obtained from it showed maxima at 280 m$\mu$. Difference spectra between the alkaline (pH 12) and neutral solutions (pH 6) showed maxima at 298 m$\mu$. The ratio between $\Delta a_{298}$ of the difference spectrum and $a_{280}$ of the neutral spectrum was 0.61 for compound 6, and 1.44 for the aglycone obtained from it. The infrared spectrum of the aglycone from compound 6 was determined as a film deposited on a salt plate and is shown in Fig. 5.

The ultraviolet spectrum of compound 7 was determined in methanol and found to have a maximum at 278 m$\mu$. The difference spectrum between the alkaline and neutral solution of compound 7 was similar to that of compound 6 in that it showed a maximum at 298 m$\mu$, the height of which was less than half of that to be expected from a free guaiacyl group.

Compound 7 was also enzymatically hydrolyzed exactly as described for coniferin. The test for a guaiacylglycerol nucleus was performed on compound 6, the aglycone from compound 6, and compound 7. The results were negative in each case.

In an attempt to elucidate their nature, compounds 6 and 7 and their enzyme hydrolyzates were chromatographed with solvent *j*. After spraying with spray *b*, compound 6 showed an orange-yellow spot, $R_f = 0$. The hydrolyzate showed a strong orange-pink spot, $R_f$ 0.20, and a very weak pink spot, $R_f$ 0.70 which corresponded in $R_f$ and color reaction with pinoresinol. The hydrolyzate of the sample of compound 6 obtained from the water-soluble portion of the butanol extract showed, in addition to the same spots, a very weak purple spot, $R_f$ 0.50, corresponding to coniferyl alcohol. Compound 7 showed an orange-yellow spot, $R_f$ 0.05, its hydrolyzate, an orange-yellow spot, $R_f$ 0.10, and a weak pink spot, $R_f$ 0.40, which corresponded in $R_f$ and color reaction with dehydrodiconiferyl alcohol.

***Extractives of the Sapwood***

Since characterization of compound 6 was hindered by the small amount available from the cambial zone,

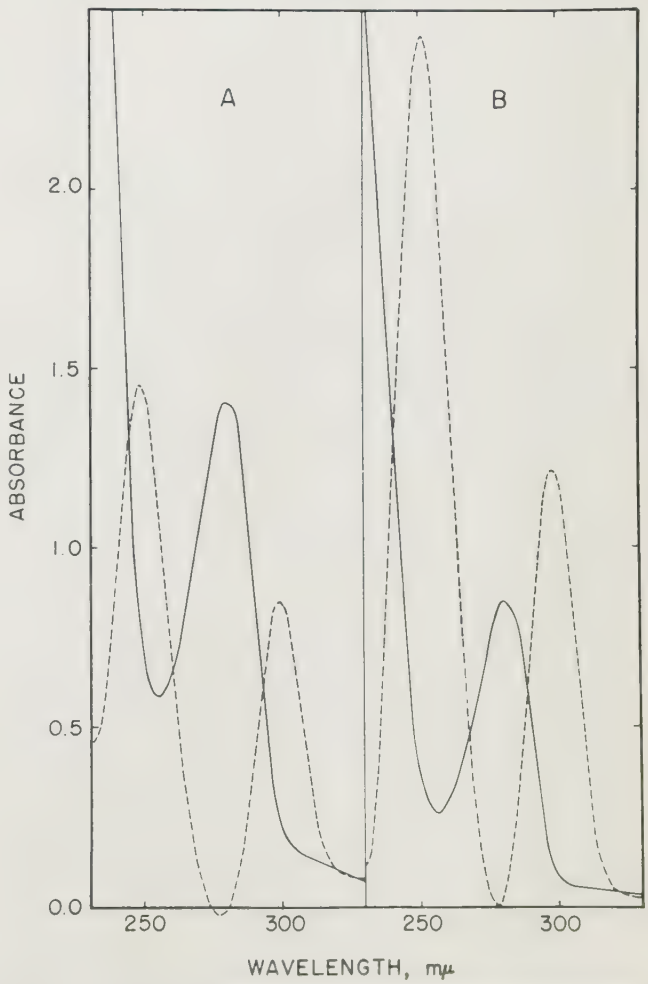



*Fig. 6. Ultraviolet absorption spectrum of phenolic glucoside (compound 6), (A) before, and (B) after hydrolysis*

— pH 6; - - - difference spectrum (pH 12 vs. pH 6).

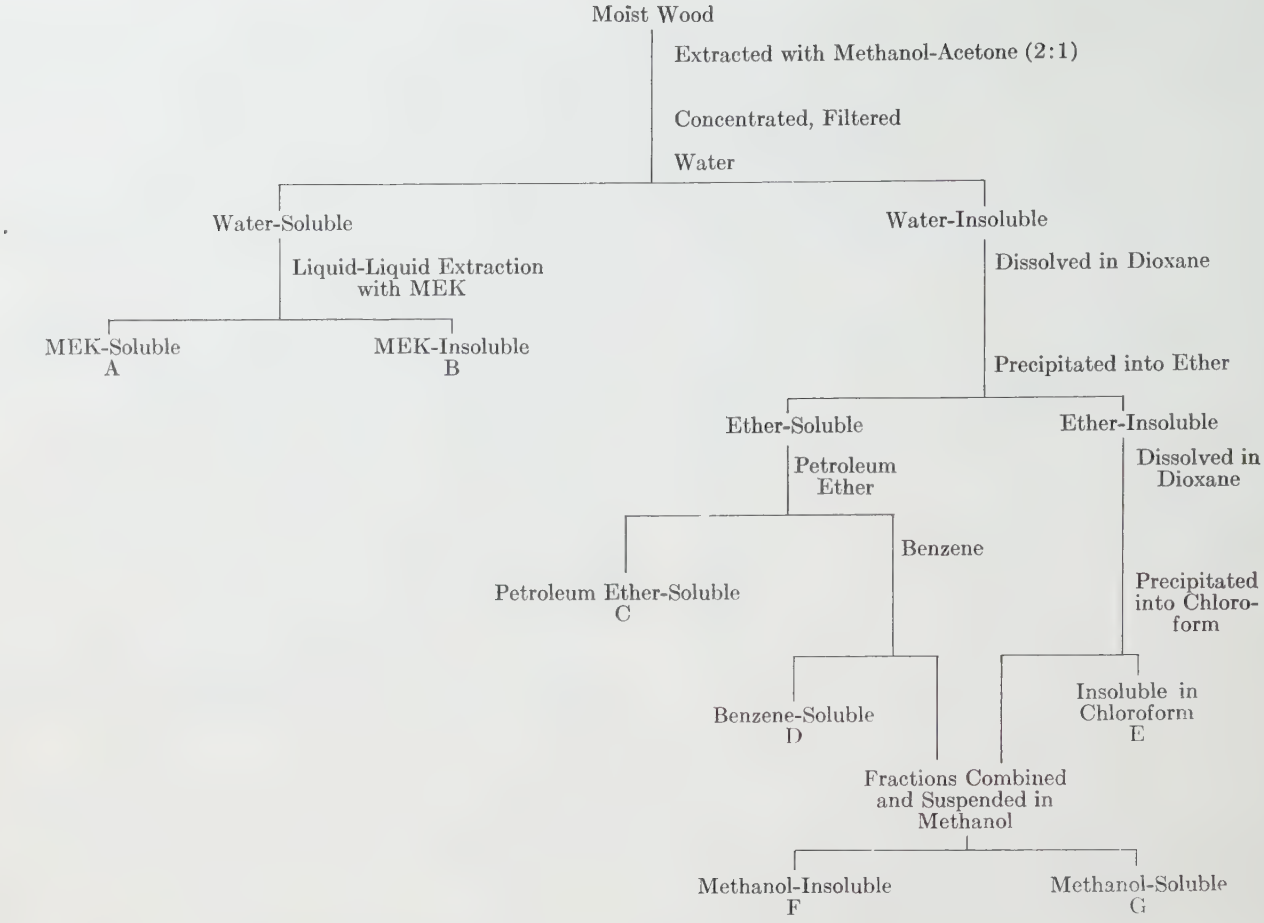



***Fig. 7. Separation scheme for Western Hemlock Sapwood extractives***

sapwood, heartwood, and bark were examined to see if this compound was also present there and thus more readily available. Also, it appeared desirable to determine the extent to which other cambial constituents were present in the wood and bark. Shavings of sapwood, heartwood, and inner bark from a newly felled fifty-year-old tree were extracted with several portions of warm methanol-benzene (2:1). The extracts were concentrated in vacuum and spotted, along with a corresponding cambial extract, on Whatman no. 1 paper. The sheets were submitted to two-dimensional chromatography with solvent systems *a* and *i*, dried, and sprayed with diazotized sulfanilic acid (reagent *b*). The chromatograms (Fig. 3) indicated the presence of compound 6 in both sapwood and inner bark.

Extractive fractions were prepared from the wood as follows: Forty-five kilograms of fresh sapwood (56% moisture content) was chipped, further reduced in a hammermill, and screened to pass a 20-mesh sieve. The wood meal was immediately placed in a stainless steel tank and immersed in a mixture of methanol-acetone (2:1 by volume). After standing for 24 hr., the supernatant liquid was drained and replaced with fresh solvent. This procedure was repeated three times. The extracts were combined and reduced to about 2 liters in a natural circulating evaporator. Due to the considerable amount of water present in the wood, the extract at this point consisted of an aqueous suspension of insoluble and soluble materials.

*Fractionation of Extractives.* The mixture was centrifuged, the aqueous phase being decanted, and the insoluble material was extracted with an additional liter of warm water. The water extracts were combined and evaporated to about 600 ml. in a rotating film evaporator. The water extract was liquid-liquid extracted with five 500-ml. portions of water-saturated methyl ethyl ketone. The MEK-soluble material (fraction A) weighed 4.50 g. The water-soluble, MEK-insoluble fraction (B) contained 67.6 g. of solids.

The water-insoluble materials were dried *in vacuo* at 40°C. for 48 hr., and dissolved in about 750 ml. of purified dioxane. The dioxane solution was filtered and poured with stirring into 5 liters of ether. The precipitated Brauns "native lignin" was washed with warm ether until no additional ether-soluble material was removed. The native lignin fraction was further purified by dissolving in dioxane and reprecipitating into chloroform. After washing with benzene, and then petroleum ether, it was dried *in vacuo* (fraction E). The chloroform- and ether-soluble filtrates were combined, evaporated to dryness, and extracted with petroleum ether (40–60°C., boiling range) and benzene to give fractions C and D, respectively. The benzene-insoluble material was taken up in 200 ml. of hot methanol, cooled, and allowed to stand for 48 hr. An insoluble white precipitate was filtered off (fraction F) and the filtrate evaporated to dryness (fraction G). An outline of the separation is shown in Fig. 7. The yields of the various fractions and their chemical nature are shown in Table V.

*Brauns "Native Lignin."* The infrared spectrum of the crude "native lignin," prior to precipitation into

**Table V. Yield and Nature of Extractives from Hemlock Sapwood**

Percentages based on O.D. weight of wood

| *Fraction* | *Nature of fraction* | *Yield, %* |
|---|---|---|
| A | Water-soluble phenols, fumaric acid | 0.023 |
| B | Sugars, compounds 6 and 7, polymeric carbohydrates, sequoyitol, pinitol | 0.341 |
| C | Wax, fat, and resin acids | 0.237 |
| D | Brown resinous wax | 0.101 |
| E | Brauns "native lignin" | 1.100 |
| F | Conidendrin | 0.052 |
| G | Hydroxymatairesinol and other lignans | 0.342 |
| | Sum | 2.196 |

chloroform, showed a moderately strong band at 1750 cm.$^{-1}$ indicative of a six-membered ring lactone. Two-dimensional paper chromatography in solvent systems *a–i* and *b–i* indicated the presence of appreciable amounts of the lactone subsequently identified as hydroxymatairesinol. It was found that this and other lignanlike contaminants could be largely removed by precipitating a dioxane solution of the crude "native lignin" into chloroform. These reprecipitations were repeated until the infrared spectrum of the chloroform-insoluble "native lignin" showed the substantial absence of absorption at 1750 cm.$^{-1}$. The product obtained in this way gave a magenta coloration when treated with phloroglucinol and hydrochloric acid in ethanol, a positive test for the presence of a guaiacylglycerol nucleus, and a positive test for an alpha-hydroxy-guaiacyl nucleus. Analyses indicated 64.00% carbon, 5.89% hydrogen, 14.61% methoxyl, 4.50% phenolic hydroxyl (difference spectrum) (*23*), and 1.43% phenolic hydroxyl *para* to a free alpha-hydroxyl (determined by the monochloroquinoneimide method of Gierer) (*24*). Other studies on this "native lignin" fraction are described elsewhere (*25*).

*Conidendrin.* Fraction F was dissolved in a minimal amount of boiling acetone, filtered, and allowed to cool. A white crystalline product, m.p. 254–255°C., $[\alpha]_D^{22}$ −51.6 (*c*, 1.865 in acetone), was obtained. The infrared spectrum was measured as a paraffin oil mull and found to be identical with that of *l*-α-conidendrin. A mixed m.p. with an authentic sample of conidendrin was undepressed. Approximately 94% of fraction F was recovered as crystalline conidendrin. The remainder of the fraction was a dark resinous material, apparently polymeric, which did not separate into resolvable components upon paper chromatography in a variety of solvent systems.

*Hydroxymatairesinol.* Fraction G was a pale-lemon colored resin. Two-dimensional paper chromatography in solvent systems *a* and *i*, *b* and *i*, or *c* and *h* indicated that this fraction consisted primarily of a phenol giving a bright-orange color with spray reagent *b*. Attempts were made to obtain the compound in a pure state by recrystallization from a variety of solvents. The only solvents which offered promise were hot water and benzene. The product obtained upon cooling a saturated, hot water solution of fraction G was a white, amorphous powder upon drying, but paper chromatography indicated that it was still not a pure compound. Fraction G was then suspended in a large volume of boiling benzene. After filtering and cooling, the mixture deposited white "crystals" upon standing. These were filtered off and dried *in vacuo*, but the crystals turned into a white powder upon removal of the last traces of benzene. The product obtained after a second "recrystallization" from benzene was found to be relatively free of the other phenolic constituents present in fraction G. The compound was not fluorescent and had an $R_f$ of 0.90, 0.13, 0.03, and 0.85 in solvent systems *a*, *b*, *c*, and *i*, respectively, on Whatman no. 1 paper.

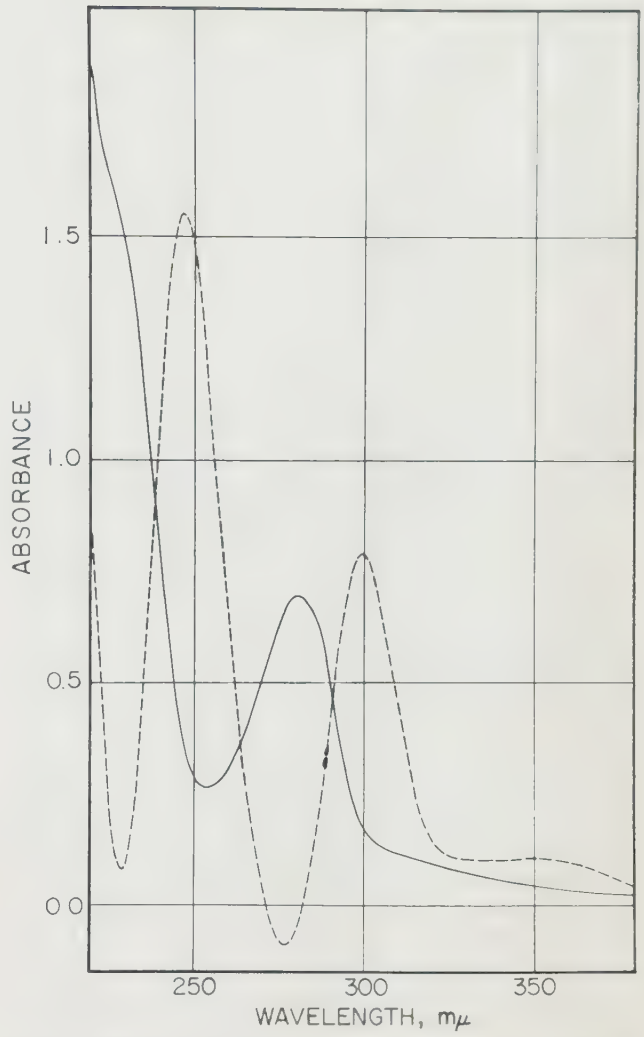



***Fig. 8. Ultraviolet absorption spectrum of hydroxymatairesinol***

— in ethanol; - - - difference spectrum (pH 12 vs. pH 6).

*Anal.* Calcd. for $C_{20}H_{22}O_7$: C, 64.16; H, 5.92; $OCH_3$, 16.58; phenolic OH, 9.09. Found: C, 64.51; H, 5.83; $OCH_3$, 16.46; phenolic OH, 8.90 (*23*).

The product was very slightly soluble in boiling benzene, ether or water, moderately soluble in chloroform, and readily soluble in polar solvents such as acetone, methanol, or ethanol. The product softened at about 95°C., but did not give a true m.p. It was levo rotary, $[\alpha]_D^{22}$ −10.4 (*c*, 4.0 in tetrahydrofuran), $[\alpha]_D^{22}$ −8.3 (*c*, 4.0 in 95% ethanol). The ultraviolet neutral, alkaline, and difference spectra were determined in ethanol, indicating the presence of an unconjugated phenolic nucleus (Fig. 8). The infrared spectrum was measured as a film from chloroform. The strong peak at 1745 cm.$^{-1}$ indicated the presence of a lactone ring. The test for an alpha-hydroxyl-guaiacyl nucleus was positive. Careful examination of the chromatograms of the coupled phenol obtained during this test indicated the presence of a second, pink

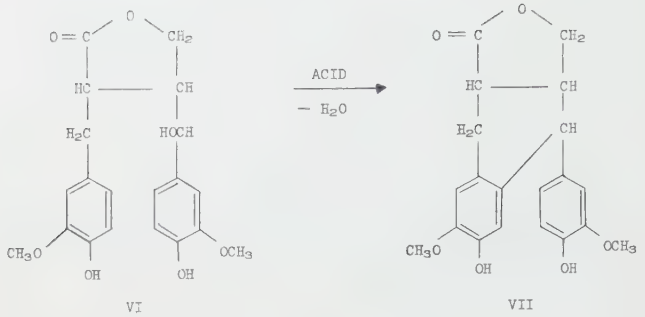



*Fig. 9*

product of lower $R_f$ than the cleaved, coupled guaiacyl nucleus. This suggested that the product was a dimer containing one guaiacyl nucleus with an alpha-hydroxy group and another attached to a —$CH_2$— group.

The acetate derivative was prepared by the acetic anhydride–pyridine procedure. The product resisted crystallization from a variety of solvents. It could be obtained as a white powder, m.p. 90–92°C., by precipitation of a methanol or chloroform solution into benzene–petroleum ether (1:2). The infrared spectrum (Nujol mull) showed bands at 1760, 1750, and 1735 cm.$^{-1}$ indicating the presence of phenolic acetoxy carbonyl, lactone carbonyl, and unconjugated aliphatic acetoxy carbonyl groups, respectively, and confirmed the alpha-hydroxyguaiacyl test. The test for a guaiacylglycerol grouping by acidolysis in dioxane-HCl was negative. However, the concentrated dioxane solution after the reaction contained white crystals, m.p. 255°C. These were characterized as conidendrin (VII), Fig. 9, a mixed m.p. with an authentic sample being undepressed. Near quantitative conversion of the product to conidendrin was achieved by suspending two grams in 15 cc. of formic acid and allowing the mixture to stand overnight. The crystals were filtered off, washed with methanol, and dried, m.p. and mixed m.p. 254–255°C., $[\alpha]_D^{22}$ −52.4 (*c*, 2.0 in acetone). The physical properties and acidic condensation to conidendrin establish the compound to be *l*-hydroxymatairesinol (VI) (*26*), lit., $[\alpha]_D^{25}$ −11.0° in tetrahydrofuran, −6.3 in ethanol). Approximately 85% of fraction G consists of this compound.

Since hydroxymatairesinol was found to be present in the hemlock wood in a ratio of about 5:1 to that of conidendrin, it was thought to be of interest to determine whether conidendrin in sulfite spent liquor might, in part, be derived from hydroxymatairesinol. One gram of hydroxymatairesinol was dissolved in 20 cc. of methanol and adsorbed on 20 g. of fibrous alpha-cellulose. After evaporation of the methanol, the pulp and 100 cc. of ammonia-base sulfite cooking liquor (0.75% combined and 6.63% free sulfur dioxide) were placed in a stainless steel autoclave, and heated for 5 hr. at 135°C. in an oil bath. The autoclave was then quickly cooled and the contents filtered, the pulp being washed with 200 ml. water and then dried. The dried pulp was extracted with acetone, and the aqueous filtrates were extracted with ether. The solvent-soluble fractions were combined, evaporated to dryness, and dissolved in 10 cc. of hot methanol. Upon cooling, crystals of conidendrin, m.p. and mixed m.p. 253–255°C. after recrystallization, were obtained in about 75% yield of the starting product. Chromatography of the filtrate demonstrated the absence of unreacted hydroxymatairesinol. Traces of vanillic acid, $R_f$ 0.20 and 0.54 in solvent systems *b* and *i*, were present, indicating that some degradation had taken place during the conversion of hydroxymatairesinol to conidendrin.

Sulfite spent liquor from a calcium base sulfite cook of Western hemlock wood was liquid-liquid extracted with ether. The ether extract and the ether-insoluble, aqueous fraction were chromatographed two-dimensionally in systems *a–i* and *b–i*. Neither hydroxymatairesinol nor any other substances, giving an orange color with spray reagent *b* (except vanillic acid) was present.

*Oxomatairesinol.* After long exposure (six months) of hydroxymatairesinol to air, or upon long standing in ethanolic solution, a compound, $R_f$ 0.09 and 0.86 in solvent systems *c* and *i*, was slowly formed which gave a bright bluish purple fluorescence under ultraviolet light. The compound no longer gave an orange color with spray reagent *b*. Instead it gave a weak pink color. This suggested that the alpha-hydroxyl group had oxidized to a keto group, i.e., that oxomatairesinol (*26*) had formed. Chromatograms of fractions D and G both show spots of $R_f$, fluorescence, and color reactions identical with that of the oxidized hydroxymatairesinol, so it appears that oxomatairesinol is present in hemlock wood.

*Pinoresinol.* The benzene-soluble material remaining after fractional precipitation of the hydroxymatairesinol (fraction G-1) was submitted to one- and two-dimensional chromatography. A compound giving an intense pink-colored spot (spray reagent *b*), $R_f$ 0.92, 0.77, 0.54, and 0.60 in solvent systems *a*, *b*, *c*, and *i*, respectively, was detected. The $R_f$ and color reactions were identical with those of an authentic sample of pinoresinol, kindly supplied by Dr. H. Erdtman of the Royal Institute of Technology, Stockholm. From the intensity of the spot, it is estimated the pinoresinol amounts to about 3% of fraction G, i.e., 0.009% of the wood.

*Dehydrodiconiferyl Alcohol.* Two-dimensional chromatography of fractions D and G-1 (see preceding paragraph) showed the presence of a very small amount of a compound, $R_f$ 0.11 and 0.66 in solvent systems *b* and *i*, which gave a weak pink color with spray reagent *b*. The $R_f$ and color reactions were identical with those of an authentic sample of dehydrodiconiferyl alcohol which was kindly supplied by Dr. Karl Freudenberg, Heidelberg, Germany. A second closely related compound, dehydrodiconiferylaldehyde (*27*), which is formed upon standing of dehydrodiconiferyl alcohol in ethanolic solution or long exposure to air, was observed in fraction G-1. This occurs at $R_f$ 0.06 and 0.35 to 0.45 in solvent systems *b* and *i* and can be readily detected by the distinctive magenta coloration produced by spray reagent *e*.

*Vanillic Acid.* Vanillic acid was detected in fractions D and G by $R_f$ and color reaction. Vanillic acid gives a bright-orange color with diazotized sulfanilic acid (spray reagent *b*) and is not easily distinguished chromatographically from hydroxymatairesinol. The only satisfactory solvent system found to be useful was two-dimensional chromatography with solvent system *b* followed by system *i*.

*Vanillin and Coniferylaldehyde.* Vanillin and coniferylaldehyde were detected in fraction D by two-dimensional paper chromatography with solvent system

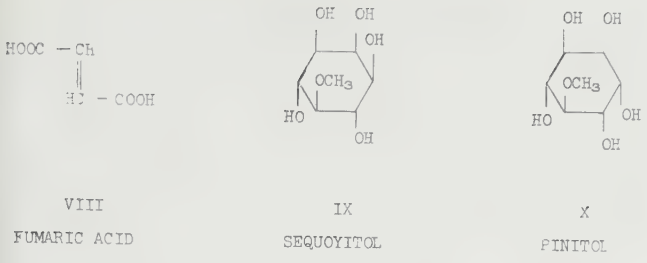



*Fig. 10*

*c* followed by solvent system *h* or *i*. Ultraviolet fluorescence after fuming with ammonia (dark purple and greenish yellow), $R_f$ values in *c* and *i* (0.70 and 0.50 for vanillin, 0.44 and 0.38 for coniferylaldehyde), and color reactions with spray *e* (pink and magenta) confirmed the presence of vanillin and coniferylaldehyde.

*Ferulic Acid.* Ferulic acid was detected in fraction G-1 (and traces in fraction D) by the typical pair-wise arrangement of spots due to the *trans* and *cis* isomers in solvent system *i* ($R_f$ 0.32 and 0.62), ultraviolet fluorescence (bright blue after exposure to ammonia fumes), and purplish red coloration with diazotized sulfanilic acid (reagent *b*).

*Fumaric Acid.* Fraction A was evaporated down to a volume of 150 ml. and placed in the icebox. After several days, a light-brown precipitate deposited. This was filtered off and the filtrate placed back in the icebox. After a week, an additional crop of crystals were obtained. These were combined with the first crop, suspended in 20 ml. water and heated to boiling. The water-soluble part was decanted from a brown waxy insoluble portion, cooled to about 60°C., treated with decolorizing charcoal, filtered, and allowed to stand overnight at room temperature. About 150 mg. of white hexagonal crystals, m.p. 285°C. (with decomposition) were obtained.

*Anal.* Calcd. for $C_4H_4O_4$ (116.1): C, 41.39; H, 3.47; equivalent weight, 58.0. Found: C, 41.79; H, 3.89; equivalent weight, 59.1. The infrared spectrum was identical with that of fumaric acid (VIII), Fig. 10, (reported m.p., 286–287°C.).

*Guaiacylglycerol-β-coniferylether.* The combined filtrates from the fumaric acid precipitations were concentrated and stirred into water. The mixture was filtered, and the water-soluble fraction was extracted with ether. The extracted water layer was then concentrated to about 20 ml. An aliquot was evaporated to dryness and tested for the presence of a guaiacylglycerol nucleus. A positive result was obtained. All efforts to obtain a crystalline product from the fraction were unsuccessful, however. Paper chromatography indicated the presence of hydroxymatairesinol, compounds 6 and 7, and a compound giving an orange color with diazotized sulfanilic acid (reagent *b*) which was indicative of an alpha-hydroxyguaiacyl compound. The $R_f$ of this compound in benzene saturated with water (0.70) is comparable to that reported (*10*) by Freudenberg (0.68) for guaiacylglycerin-β-coniferylether, but since an authentic sample was not available, the identification is only tentative. Crude fraction G also gives a weak test for the presence of a guaiacylglycerol derivative and shows a spot at $R_f$ 0.70 giving an orange color with spray reagent *b*.

*Catechin and Leucocyanidin.* Two-dimensional chromatography of fraction A and B in solvent systems *a* and *i* demonstrated the presence of compounds corresponding in $R_f$ and color reactions (*28*) to catechin (0.68, 0.46), epicatechin (0.56, 0.37), gallocatechin (0.48, 0.40), epigallocatechin (0.40, 0.33), and four leucocyanidins (3,4,5,7-tetrahydroxy-flavan-3,4-diol) at $R_f$'s 0.40 and 0.55 (compound 19), 0.45 and 0.52 (compound 20), 0.38 and 0.41 (compound 21), and 0.35 and 0.47 (compound 22). Hydrolysis of the crude mixture (fraction B) by the procedure of Bate-Smith (*29*) yielded cyanidin and traces of delphinidin.

*Sugars.* The water-soluble fraction (B), about 30% solids, was poured into methanol to precipitate any polymeric carbohydrates which might interfere in subsequent separations. Quantitative paper chromatography (*17*) of the 80% methanol-soluble solids indicated the presence of 0.65% (based on the O.D. weight of the fraction) xylose, 0.48% arabinose, 1.1% mannose, 4.5% glucose, and 0.69% galactose. A spot at $R_f$ 0.37 in solvent system *a* corresponding to rhamnose was also present, but the amount was not determined.

*Quinic and Shikimic Acids.* A sample of methanol-soluble material from fraction B was chromatographed with solvent *a* and sprayed with reagent *h*. Two spots, $R_f$ 0.20 and 0.40, were observed. These corresponded with quinic acid and shikimic acid.

*Sequoyitol.* The methanol-soluble material from fraction B was deionized by passing it through a column of IR-120 ($H^+$) resin. It was subsequently passed through a column of Dowex-3 ($AcO^-$) to remove carboxylic acids. The neutral effluent and aqueous washings were concentrated *in vacuo* to remove methanol. This solution (150 ml.) was extracted with three, 300-ml. portions of *n*-butanol. Upon standing, crystals separated from both the butanol extract and from the butanol-extracted aqueous fraction. The crystals were filtered off from the butanol-soluble extract which was subsequently used for the isolation of compounds 6 and 7 as described previously.

The crystals in the aqueous fraction were filtered off and, since they were found to be identical with those deposited from the butanol extract, the two crystalline fractions were combined. The crystalline material was then dissolved in a minimum amount of 50% methanol, treated with decolorizing char, filtered, and cooled to give 2.5 g. white plates, m.p. 239–240°C. (after one additional recrystallization). The $R_f$ in solvent system *f* and the infrared spectrum (Fig. 5) were identical with those of an authentic sample of sequoyitol (IX) kindly furnished by Dr. Laurens Anderson of the Department of Biochemistry, University of Wisconsin. A mixed m.p. with the authentic sample was undepressed, further indicating the identity of the two samples. The acetate derivative was prepared by the acetic anhydride-pyridine method and recrystallized from ethanol to give flat white needles, m.p. 199–200°C. [lit. (*9*) for sequoyitol pentaacetate, 200–202°C.].

*Pinitol.* After sequoyitol was separated from the butanol-insoluble, aqueous fraction, it was evaporated to about 50% solids and allowed to stand for about six months. During this time, crystallization slowly occurred in the sirupy mixture. The crystals (3.5 g.) were separated by filtration and washed with a mixture of ethanol and dilute hydrochloric acid (90:10). White crystals, m.p. 185–186°C., were obtained after recrys-

tallization from 80% ethanol. The infrared spectrum (Fig. 5) was determined as a paraffin (Nujol) mull and found to be identical in every respect with the spectrum (*30*) of D-pinitol (X) (m.p. 186°C.).

## RESULTS

Isolation of phenolic constituents from cambial material proved to be exceedingly difficult owing to the complexity of the mixture of compounds present therein. This is readily illustrated by a two-dimensional chromatogram (Fig. 3) of the ethanol extract of the cambial material. The separation of substances by the conventional techniques of fractional crystallization, etc., therefore, was not, in general, practicable, chromatographic techniques being largely used.

As a result of these studies the following extractive substances were detected in Western hemlock cambium: quinic acid, shikimic acid, sucrose, fructose, glucose, coniferin, leucocyanidin (3′,4′,5,7-tetrahydroxyflavan-3,4-diol), catechin, epicatechin, and depsides of the following acids: caffeic-shikimic, ferulic-shikimic, ferulic-quinic, and *p*-coumaric-shikimic. A fifth depside was tentatively identified as *p*-coumaryl-quinic acid. In addition to these compounds, several glycosides of lignanlike compounds were detected, but their structure has not yet been completely elucidated.

Coniferin has been previously reported in the cambia of several coniferous genera (*31*), as have quinic acid, shikimic acid, glucose, fructose, and sucrose (*5*), but this is the first reported detection of these compounds in *Tsuga*. Leucocyanidin, catechin, and epicatechin have been found to be of nearly ubiquitous occurrence in conifers (*28*). Although the depside chlorogenic acid and its related isomers are widely distributed in the plant kingdom, this is the first reported occurrence of cinnamic depsides in the conifers. As far as can be determined, depsides of caffeic-shikimic, ferulic-shikimic, and *p*-coumaric–shikimic acids have not been previously reported from any source.

The glycosides of the lignanlike compounds (termed compounds 6 and 7 on chromatograms and in the experimental section) were particularly difficult to isolate and characterize. This was, in the main, due to their solubility in the same solvents as the sugar and depside components of the cambium and lack of solubility in solvents suitable for separation from these latter components. Compound 6 was isolated chromatographically. It gave an orange color with diazotized sulfanilic acid indicating the presence of an alpha-hydroxyguaiacye nucleus. The ultraviolet neutral and alkaline difference spectra (Fig. 6), however, showed only half of the phenolic hydroxyl content to be expected from a free guaiacyl group. This suggested that the compound was a dimer containing one free guaiacyl group. This was confirmed by enzymatic or acidic hydrolysis of compound 6 from which glucose was obtained. The ultraviolet neutral and difference spectrum of the resultant aglycone now gave about twice the phenolic hydroxyl content previously observed in the unhydrolyzed compound. Chromatography of the hydrolyzed mixture showed, in addition to glucose and the principal spot from the aglycone, two weak spots. These corresponded in color reactions and $R_f$ to coniferyl alcohol and pinoresinol. Since compound 6 was found to have virtually the same $R_f$ in the various chromatographic solvent systems employed as that of coniferin, it is quite likely that a small amount of coniferin was present as a contaminant in compound 6 and was the source of the coniferyl alcohol in the enzymatic hydrolyzate. Presumably the pinoresinol came from a similar source, e.g., a pinoresinol glucoside with similar $R_f$ as compound 6 and also present as a contaminant. Because of the means of isolation of compound 6 (see Experimental), it is possible that the pinoresinol glucoside is a sapwood constituent rather than a true cambial constituent. Further work will be required to substantiate this point. The hydrolysis experiments were repeated several times in several ways, always with the same results, so it does not appear that the pinoresinol is an artifact due to dehydrogenative coupling of coniferyl alcohol.

The ultraviolet spectrum of the aglycone of compound 6 is typical for that of unsubstituted unconjugated guaiacyl groups and does not suggest that the two guaiacyl groups are joined through the nucleus in a diphenyl-type linkage. The infrared spectrum of the aglycone indicates the presence of an unconjugated carbonyl group, probably ketonic, since the compound gives a weak color reaction with 2,4-dinitrophenylhydrazine. The orange-pink coloration of the aglycone with diazotized sulfanilic acid suggests that the other guaiacyl nucleus is attached to a $—CH_2—$ linkage. From the properties of compound 6, it is evident that it has not been previously described in the literature. Sufficient data are not yet at hand to assign a structure to the compound.

Compound 7 gave the same orange color reaction with diazotized sulfanilic acid, and the same ultraviolet neutral and difference spectra, as those of compound 6, indicating the presence of an alpha-hydroxyguaiacyl nucleus, and less than half the phenolic hydroxy content to be expected from a free guaiacyl group. Chromatograms of the enzymatic hydrolyzate of compound 7 with carbon tetrachloride-dimethylformamide showed a spot similar in $R_f$ and color reaction to guaiacylglycerol-β-coniferylether. A test for the presence of the guaiacylglycerol nucleus, however, was negative. In addition to this spot, chromatograms showed a weak pink spot that corresponded in $R_f$ and color reaction to dehydrodiconiferyl alcohol. It would appear, therefore, that a glucoside of this substance was present as a contaminant in compound 7. The absence of coniferyl alcohol in the hydrolyzate of compound 7, and of dehydrodiconiferyl alcohol in the hydrolyzate of compound 6 is further evidence that the dimers were not artifacts derived from coniferyl alcohol: For a solution of coniferyl alcohol, upon aging, always showed the spots of both pinoresinol *and* dehydrodiconiferyl alcohol. The amount of compound 7 present was less than that of compound 6 and insufficient for further characterization of compound 7 or its components.

None of the chromatograms of cambial material, except those of enzyme-treated material, showed spots of coniferyl alcohol, dehydrodiconiferyl alcohol or pinoresinol. A spot corresponding to guaiacylglycerol-β-conferyl ether was not detected under any condition.

The constituents of hemlock sapwood were also examined in order to determine which of the cambial components were present or absent. Since this investigation was primarily concerned with lignin biogenesis, no attempt was made to identify all of the constituents

of the waxy or carbohydrate fractions. The most noticeable differences between cambial phenolic constituents and those of the sapwood, especially as revealed by chromatograms (Fig. 3), can be summarized as follows: (a) cambial cinnamic depsides are absent in the sapwood; (b) cambial phenolic glucosides are present only in trace amounts in the sapwood; and (c) the predominant phenolic constituents in the sapwood are lignans, none of which were found in the cambium as aglycones.

The largest extractive fraction in the sapwood, in terms of yield, is Brauns "native lignin." This material has been the subject of several previous investigations (*28*, *32*). Although this native lignin has a number of properties similar to those of whole wood lignin, certain features of the infrared spectrum (*28*) and the high phenolic hydroxyl and para hydroxyl benzyl alcohol content indicate that this material is not identical with whole wood lignin.

The phenolic compound present in the largest amount, next to that of the native lignin fraction, was characterized as hydroxymatairesinol. This compound was first isolated several years ago from spruce (*Picea excelsa*) wood by Freudenberg and Knof (*26*) and the present report extends the reported occurrence of this compound to the *Tsuga* genus. Conidendrin, previously reported in Western hemlock by Brauns (*33*), was isolated during the present study from sapwood in a yield of about 0.05%. The yield from heartwood is somewhat higher, i.e., 0.15–0.20%, but is still considerably less than that obtainable from spent liquor from sulfite pulping of Western hemlock (about 0.5%, based on unpulped wood). This suggested that part of the conidendrin obtained from sulfite liquor is not derived from wood as such but is formed during the cook. It was found that hydroxymatairesinol, which occurs in spruce and hemlock in three to five times the amount of conidendrin, is readily converted to conidendrin under sulfite cooking conditions. Furthermore, there is no trace of hydroxymatairesinol in spent liquor of hemlock or spruce wood. There is little doubt that the conidendrin in spent sulfite liquor is derived in large part from the condensation of hydroxymatairesinol. It is interesting to note that the original investigators (*34*, *35*) of "sulfite liquor lactone," as conidendrin was termed at that time, believed that the compound was formed during sulfite cooking. Subsequent workers (*33*) rejected this hypothesis when it was found that conidendrin could be isolated directly from wood. Our results indicate that both conclusions were correct, e.g., part of the conidendrin is derived from that originally present in the wood and part is formed during the cook by condensation of hydroxymatairesinol.

Other phenolic compounds detected in the wood were coniferylaldehyde and vaniilin, both of which have been found (*36*) in the heartwoods of other species. Pinoresinol was detected chromatographically as was dehydrodiconiferyl alcohol. The latter compound was present only in trace amounts in the sapwood and could not be detected at all in the heartwood. Traces of at least six other lignanlike compounds, in addition to oxomatairesinol, dehydrodiconiferylaldehyde, and vanillic acid, were detected on two-dimensional chromatograms of both sapwood and heartwood extracts, but these were not characterized.

Catechin, epicatechin, gallocatechin, epigallocatechin, and four leucocyanidins were detected in very small amounts in sapwood. All these compounds, with the possible exception of traces of catechin, were absent in the heartwood. A polymeric material ("phlobaphene") with low $R_f$ in both organic and aqueous solvent systems, but which has color reactions similar to those of catechin and leucocyanidin, is present in the heartwood. This appears to be a condensation product of the catechins and leucocyanidins (*28*).

Pinitol and sequoyitol were isolated from the sapwood. Pinitol has been previously found in various pines (*9*) and both pinitol and sequoyitol have been found in redwood (*37*). Neither compound has been previously isolated from Western hemlock. The isolation of fumaric acid is somewhat surprising since this acid has not previously been reported as present in wood. Chromatograms indicated the presence of other simple acids, one of which may be succinic acid. These other acids were not present in sufficient amounts for characterization.

## DISCUSSION

The role of coniferin as an intermediate in lignin biosynthesis has been the subject of considerable study. The importance of the shikimic acid pathway for the biosynthesis of phenylpropane units from glucose in higher plants is reasonably well established (*38*). Since it is not known, however, where coniferin is formed in the tree, it cannot yet be decided whether the glucose, sucrose, and shikimic acid present in the cambium play a role in coniferin synthesis. These same sugars, shikimic acid, and especially quinic acid, are also present in large amounts in the needles and presumably in the sap stream.

The physiological function in the plant of the widely occurring cinnamic esters such as chlorogenic acid is not yet clearly understood. The occurrence in hemlock cambium of shikimic acid esters of *p*-coumaric, caffeic, and ferulic acids is of interest because all the components of these depsides are known from radioactive tracer experiments to be efficient precursors of the phenylpropane groups of lignin (*39*). Chromatograms of cambial extracts from other coniferous species such as *Abies* and *Pinus*, however, failed to show any traces of these readily detected, highly fluorescent depsides. It must be concluded, therefore, that their occurrence in cambial extracts from Western hemlock, in which they were always present, is a peculiarity of this species, and that they are not involved in lignin formation.

The occurrence of the glucosides of lignanlike compounds, like that of the various catechin derivatives and the leucocyanidins is not restricted to the cambial zone. These substances were also found in the sapwood and the inner bark. It appears most likely that these compounds are intermediates in the formation of extractive components and are not involved in lignin biosynthesis.

Perhaps the most important result of this investigation was the finding that Freudenberg's proposed intermediates in lignin formation (I–IV) were absent in the cambium. Their presence in the wood, with the exception of II and III was also doubtful. On the basis of this result, and the detection of phenolic glucosides, one might be tempted to reject Freudenberg's hypothesis and suggest instead that lignification pro-

ceeds *via* glucoside intermediates. However, it is possible, as Freudenberg has suggested (*4*), that intermediates I–IV are of such a transitory nature, that they cannot be detected. It is probable that further work on the cambia of other genera, in which depsides are absent and therefore would not interfere with separation and isolation of compounds I–IV, may shed additional light on the problem of lignin biogenesis.

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# The 4-*O*-Methyl-D-glucuronic Acid Groups of Birch Xylan During Sulfate Pulping


INGEMAR CROON and BARBRO F. ENSTRÖM



**The 4-*O*-methylglucuronic content of 4-*O*-methylglucuronoxylan in birchwood (*Betula verrucosa*) has been shown to vary within wide limits (6.4–13.8%). Studies of the behavior of these uronic acid groups during ordinary sulfate pulping have shown, as expected from the work of earlier investigators, that the uronic acid content of the xylan is lower in the pulp than in the wood. However, in no case was a xylan completely free of uronic acid groups isolated from a sulfate pulp. It was possible to effect a crude fractionation of the xylan according to uronic acid content; xylan fractions varying in uronic acid content from 3–10% were isolated. As a mean value the xylan from an ordinary birch sulfate pulp (cooking yield, 52–54%) contained 3–5% uronic acid groups. When the rate of hydrolysis of the uronic acid groups is significant (at 160–170°C.) a large part of the alkali has already been consumed, and therefore the loss of uronic acid from the xylan is not complete. A complete removal of the uronic acid is, however, not necessary for a stabilization of the xylan during a sulfate cook of birchwood. One factor in the xylan stabilization at temperatures above 160°C. is probably the decrease in solubility of the xylan effected by the decrease in its uronic acid content.**


THE LARGE amount of xylan remaining in ordinary sulfate pulps, both from softwoods and hardwoods, has been explained essentially in two ways: (1) chemical linkages are formed between the xylan and the cellulose (*1*), and (2) the xylan is strongly adsorbed by the cellulose (*2*, *3*).

Lindberg, *et al.* (*4*) have shown that if the xylan is linked to lignin by phenylglycosidic linkages, the formation of a covalent linkage between the xylan and cellulose by transglycosidation [McKinney's theory (*1*)] is possible. He has also suggested a transglycosidation in connection with the alkaline hydrolysis of polysaccharides at high temperatures (around 170°C.) by the mechanism shown in Fig. 1 (*5*).

Any definite evidence for the presence of covalent bonds between hemicellulose and cellulose in sulfate pulps has, however, not yet appeared. For the present the main emphasis must therefore be concentrated on the adsorption theory in order to explain the resistance of xylan during the sulfate cook. Yllner and Enstrom studied the adsorption (under sulfate-cooking conditions) of dissolved xylan by samples of cellulose from different sources (*2*, *3*). They concluded that the transglycosidation suggested by McKinney cannot be the chief cause of the xylan stability, as the stabilization was equally effective whether or not lignin was present. Further, it was found that the amount of


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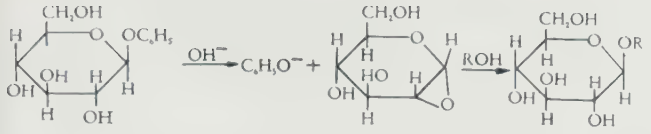

*Fig. 1. Postulated transglycosidation mechanism leading to cross-linking of wood polysaccharides*

xylan adsorbed at 170°C. was largest when the initial sodium hydroxide concentration was 2% or less. At this concentration most of the alkali was consumed during the cook. The amount of xylan adsorbed at room temperature was lower but gradually increased with temperature to about 150°C. (at a constant heating time of 3 hr.). The xylan adsorption was found to be strongly dependent on the nature of the cellulose.

Saarnio, *et al.* (*6*), reported that sulfate pulps of softwood lack uronic acids and those of hardwood contain much less uronic acid that the wood. Hamilton, *et al.* (*7*), have studied the behavior of wood hemicellulose during different pulping procedures and these results for the xylan part have been summarized in a very simple scheme (Fig. 2).

No doubt this scheme is correct as an overall picture of the direction of change during the different treatments. However, during our studies of the behavior of the xylan during sulfate pulping of birchwood (*Betula verrucosa*) we were never able to obtain a kraft pulp or to isolate a pulp xylan completely free from uronic acids (see ref. *8–9*). It was therefore decided to undertake a careful study of the change in uronic acid content of birch xylan during ordinary sulfate cooking.

## RESULTS

The structure of birch xylan has been extensively studied (*10–11*) and shown to consist of a main chain of 1,4-linked xylopyranose residues with branches of 4-*O*-methyl-D-glucuronic acid residues attached to the 2-position about every tenth xylose residue. In the native state the xylan is acetylated to an extent of 12 to 13% acetyl (*12*).

In order to obtain some knowledge of the distribution of the uronic acid groups between the xylan molecules in the holocellulose, attempts were made to fractionate the xylan according to uronic acid content. It was observed that (unlike the hemicellulose from spruce, pine, or eucalyptus) a considerable part (about 50%) of the birch xylan was precipitated from the alkaline ex-

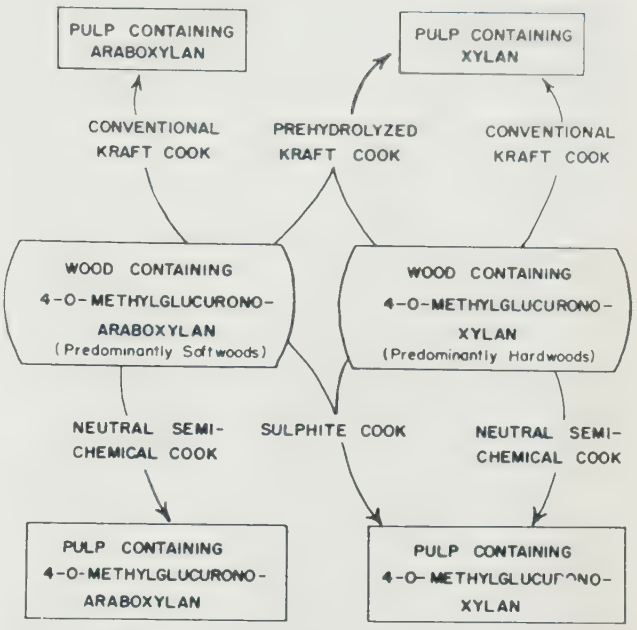


*Fig. 2. The behavior of xylan during different pulping procedures according to Hamilton, et al. (7)*

tract upon acidification with acetic acid to pH 4.5. The two fractions thus obtained showed uronic acid contents of 8.2% for the insoluble part ($\beta$) and 12.8% for the soluble part ($\gamma$). A more extensive fractional extraction with alkali showed that the most easily extracted xylan contained more uronic acid, and for each separate extraction the $\gamma$-fraction was richer in uronic acid than the $\beta$-fraction (Table I). Here a spread in uronic acid content, at least between 6.4 to 13.8%, agrees with the figures obtained by Timell and Glaudemans (*16*). The use of cetyl ammonium chloride or hydroxide as a factionating agent was proved to be unsuccessful for the present purpose (*13*).

Two birch sulfate pulps, prepared under slightly different conditions, were extracted in turn with dimethyl sulfoxide and then two aqueous sodium hydroxide solutions of 5 and 10% concentration, respectively. Dimethyl sulfoxide extraction is a very useful complement to alkaline extraction in the study of hemicelluloses in hemicellulose fractions and pulps. Treatment of the two sulfate pulps with dimethyl sulfoxide removed a considerable part of the xylan (26.4 and 23.0%, respectively) and these parts had high uronic acid contents (10.3 and 6.7%, respectively). The 5- and

**Table I. Fractional Extraction of Birch Holocellulose with Sodium Hydroxide Solutions**

| *Sample*[a] | *4-O-Methyl-D-glucuronic acid % of the xylan* | *Sugar composition* | | | | | *Yield % of the holocellulose* |
|---|---|---|---|---|---|---|---|
| | | *Galactose* | *Glucose* | *Mannose* | *Arabinose* | *Xylose* | |
| 0.1% $\beta$ | 6.7 | 0.5 | 8.9 | 0.7 | ... | 90.0 | 0.61 |
| 0.1% $\gamma$ | 9.8 | 5.2 | 0.9 | 1.1 | 1.3 | 91.5 | 1.52 |
| 0.5% $\beta$ | 7.6 | 0.6 | 2.4 | 0.6 | ... | 96.4 | 2.04 |
| 0.5% $\gamma$ | 9.9 | 6.5 | 0.6 | 1.1 | 2.2 | 89.6 | 4.82 |
| 1% $\beta$ | 6.7 | 0.6 | 1.3 | 0.7 | ... | 97.5 | 1.85 |
| 1% $\gamma$ | 13.8 | 2.5 | 0.6 | 0.8 | 0.7 | 95.5 | 3.22 |
| 2% $\beta$ | 6.4 | 0.6 | 0.6 | 1.0 | ... | 97.8 | 2.19 |
| 2% $\gamma$ | 10.4 | 1.3 | 1.0 | 1.0 | 0.9 | 95.8 | 1.61 |
| 3% $\beta$ | 7.6 | ... | 0.4 | 0.7 | ... | 98.9 | 3.80 |
| 3% $\gamma$ | 9.9 | 1.0 | 1.6 | 2.5 | 0.9 | 94.0 | 0.81 |
| 4% $\beta$ | 8.9 | 0.6 | 0.5 | 0.7 | ... | 98.1 | 1.77 |
| 4% $\gamma$ | 8.0 | 2.0 | 19.6 | 20.8 | 1.0 | 56.6 | 0.72 |
| 5% $\beta$ | 9.0 | 0.7 | 0.8 | 0.9 | ... | 97.6 | 0.80 |
| 5% $\gamma$ | ... | ... | ... | ... | ... | ... | ... |
| 10% $\beta$ | 8.0 | 0.7 | 32.7 | 7.4 | ... | 59.3 | 3.84 |
| 10% $\gamma$ | 9.2 | 0.8 | 25.3 | 45.2 | ... | 28.7 | 1.56 |

[a] 0.1% $\beta$ represents the fraction isolated by extraction with 0.1% NaOH which precipitated upon acidification with acetic acid, etc.

Table II. Extraction of the Birch Sulfate Pulps with 5 and 10% Aqueous Sodium Hydroxide Solutions

| Pulp | Fraction | Yield % of the pulp | 4-O-Methyl-D-glucuronic acid % of the xylan | Sugar composition | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | | Galactose | Glucose | Mannose | Arabinose | Xylose |
| I | α-residue | 71.9 | ... | ... | 96.6 | 0.7 | ... | 2.7 |
| | $\beta_5$ | 22.3 | 5.6 | 1.5 | 1.3 | ... | ... | 97.2 |
| | $\gamma_5$ | 2.4 | 6.7 | 2.8 | 1.9 | 2.1 | ... | 93.3 |
| | $\beta_{10}$ | 2.5 | 3.0 | 1.8 | 20.6 | 3.7 | ... | 73.9 |
| | $\gamma_{10}$ | 0.9 | 5.3 | 1.7 | 6.1 | 4.2 | ... | 88.0 |
| II | α-residue | 72.5 | ... | ... | 93.6 | 2.1 | ... | 4.3 |
| | $\beta_5$ | 22.8 | 3.9 | ... | 1.4 | ... | ... | 98.6 |
| | $\gamma_5$ | ... | ... | ... | ... | ... | ... | ... |
| | $\beta_{10}$ | 3.7 | 2.7 | Trace | 21.7 | 2.8 | ... | 75.5 |

Pulp I had the following sugar composition: glucose 71.9%, mannose 1.6%, xylose 26.5%; and pulp II: glucose 74.3%, mannose 0.7%, xylose 25.0%.

10%-sodium hydroxide extractions removed together 85 to 90% of the xylan present in the pulps, and all fractions contained uronic acid groups (Table II).

As different fractions here again had different contents of uronic acid groups, one pulp was given a more extensive extraction in order to investigate the distribution of uronic acid groups. The figures given in Table II show in this particular pulp a variation of uronic acid content in the xylan between 3.0 to 6.7%.

The fraction $\beta_5$, obtained by extraction of pulp I with 5% sodium hydroxide, was hydrolyzed, and the uronic acids were adsorbed on an anion exchange column (IR-4B, acetate form). After removal of neutral sugars, the acids were eluted with an acetic acid solution of increasing concentration. In this manner a fractionation of the uronic acids was obtained and pure 2-*O*-(4-*O*-methyl-α-D-glycopyranosyluronic acid)-D-xylopyranose could be isolated. This aldobiouronic acid was converted to its crystalline tetraacetyl derivative (14) in a yield corresponded to a uronic acid content of 3.7% in the original xylan sample which had an estimated content of 5.6%. Consequently there is no doubt that the major part of the xylan in pulp I had an average uronic acid content of about 5%.

Hamilton has shown (7) that the α-linkage between the 4-*O*-methyl-glucuronic acid and the xylose residue is sensitive to alkaline hydrolysis. A dissolved glucuronoxylan treated with an excess of alkali at temperatures above 160°C. rapidly loses its uronic acid groups to give a somewhat degraded neutral xylan. As is evident from the results discussed above, we find that significant amounts of uronic acid are still attached to the xylan in the pulp.

The same deviation in the course of a reaction in a homogeneous medium and in a heterogeneous medium was observed for the deacetylating of birch xylan with sulfite cooking acid (*15*). The dissolved xylan was deacetylated much faster than the xylan in the fiber. It seems as if the hemicellulose in the fiber is protected to a certain extent against chemical attack although it does not, at least originally, have a high degree of lateral order. It is therefore dangerous to identify the results of a reaction of a single component when dissolved with the results for the same reaction when that single component is present entangled in the solid fiber phase.

Table III. Analysis of the Different Materials Used in the Xylan Adsorption Experiments

| Material | Galactose, % | Glucose, % | Mannose, % | Arabinose, % | Xylose, % | Uronic acid, % |
|---|---|---|---|---|---|---|
| Cotton linters | ... | 99.9 | Trace | ... | Trace | ... |
| Alkali-refined birch sulfite pulp | ... | 99.0 | 0.6 | ... | 0.4 | ... |
| Birch xylan | 3.3 | 0.7 | 0.9 | 0.8 | 94.4 | 9.61 |
| Eucalyptus xylan | 2.9 | 1.9 | 0.4 | 0.5 | 94.3 | 13.7 |
| Esparto xylan | 0.5 | 2.9 | ... | 8.4 | 88.2 | ... |
| Sulfate pulp xylan from birch | 1.5 | 1.3 | ... | ... | 97.2 | 5.6 |

An examination was made of the influence of the uronic acid content of the xylan starting material on the adsorption of the xylan on linters and high alpha-cellulose sulfite pulp. Glucuronoxylan isolated from holocellulose of eucalyptus (13.7% uronic acid), birch (10.6% uronic acid), esparto grass (0% uronic acid), and from a birch sulfate pulp (5.6% uronic acid) were treated in hot alkaline solution together with the cellulose materials (Table III).

The adsorption on linters seemed to be almost independent of the uronic acid content of the starting xylan material, although the xylan with the highest content of uronic acid was adsorbed to a somewhat lower extent (Table IV). At room temperature (25°C.) a negligible quantity of xylan (irrespective of uronic acid content) was adsorbed on linters in 2% sodium hydroxide solution.

The sulfite pulp showed a much lower adsorption of xylan than the linters (Table V). This agrees with previous results (*2*). It is interesting to note the effect of alkaline concentration on the two materials. The linters adsorbed more xylan when 2% sodium hydroxide was used, in which case about 25% of the alkali remained after the cook while the sulfite pulp adsorbed more xylan when 0.5% sodium hydroxide was used, in which case all alkali was consumed. A lower alkali concentration in the end of the cook should facilitate the xylan adsorption but it may be that the swelling of the linters is so important that it will mask the alkali concentration effect. At higher alkali concentrations the adsorption will decrease (*3*).

Extraction of the adsorbed xylan and analysis of its uronic acid content showed values between 3 and 4% irrespective of whether the xylan starting material had a uronic acid content of 5.6, 10.6 or 13.7%.

## DISCUSSION

Part of the xylan (about 20%) is very easily extracted from the birchwood chips by weak alkali solutions, and this fraction dissolves early in the sulfate cooking process. The rest is presumably protected from disolution by the fiber texture. The presence of the uronic acid group in the 2-positions of xylose units in the xylan chain impedes the peeling-off reaction. Thus the xylan is not broken down to small molecules during the period of the cook when the temperature is raised.

During our studies we observed that the content of uronic acid in the xylan determines, to a great extent, the solubility of the xylan in water or weakly alkaline

Table IV. Adsorption of Xylan on Cotton Linters upon Treatment in Sodium Hydroxide Solutions at 170°C. for Three Hours

| Type of xylan | % NaOH prior to the cook | % NaOH after the cook | % NaOH consumed | Yield of pulp, % | Sugar composition, % | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | Galactose | Glucose | Mannose | Arabinose | Xylose |
| Birch xylan | 0.55 | 0 | 0.55 | 102.6 | ... | 91.4 | 1.3 | ... | 7.3 |
| | 2.05 | 0.58 | 1.47 | 100.8 | ... | 89.9 | 1.4 | ... | 8.7 |
| Eucalyptus xylan | 0.29 | 0 | 0.29 | 102.0 | ... | 94.1 | 0.9 | ... | 5.0 |
| | 1.71 | 0.45 | 1.26 | 100.4 | ... | 92.3 | 0.9 | ... | 6.8 |
| Esparto xylan | 0.39 | 0 | 0.39 | 106.6 | ... | 91.2 | 1.0 | ... | 7.8 |
| Sulfate pulp xylan from birch | 0.40 | 0 | 0.40 | 105.5 | ... | 90.5 | 0.9 | ... | 8.6 |
| | 1.86 | 0.51 | 1.35 | 104.4 | ... | 89.4 | 0.7 | ... | 9.8 |

Table V. Adsorption of Xylan on a High Alpha-Cellulose Birch Sulfite Pulp upon Treatment in Sodium Hydroxide Solutions at 170°C. for Three Hours

| Type of xylan | % NaOH prior to the cook | % NaOH after the cook | % NaOH consumed | Yield of pulp, % | Sugar composition, % | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | Galactose | Glucose | Mannose | Arabinose | Xylose |
| Birch xylan | 0.55 | 0 | 0.55 | 99.7 | ... | 92.0 | 1.5 | ... | 6.5 |
| | 2.05 | 0.49 | 1.56 | 93.6 | ... | 92.7 | 1.7 | ... | 5.7 |
| Eucalyptus xylan | 0.29 | 0 | 0.29 | 98.2 | ... | 94.5 | 1.6 | ... | 3.9 |
| | 1.77 | 0.48 | 1.29 | 91.7 | ... | 96.4 | 0.8 | ... | 2.8 |
| Esparto xylan | 1.85 | 0.28 | 1.57 | 94.1 | ... | 96.5 | 0.6 | ... | 2.9 |
| Sulfate pulp xylan from birch | 0.40 | 0 | 0.40 | 101.0 | ... | 91.6 | 0.8 | ... | 7.6 |
| | 1.93 | 0.59 | 1.34 | 91.4 | ... | 95.3 | 0.6 | ... | 4.1 |

solutions. Thus, only a small amount of sulfate-pulp xylan is soluble in water, and the main part is precipitated upon neutralization of an alkaline solution. On the other hand, about half of the xylan from birch holocellulose and almost all of the eucalyptus xylan of high uronic acid content (about 13.5%) still remain in solution after neutralization of the alkaline solutions. It is therefore possible that one factor in the xylan stabilization at temperatures above 160°C. is simply the decrease in solubility of the xylan effected by the decrease in its uronic acid content. In this connection it should also be noted that in the sulfate cook there is a complete removal of the other xylan substituents, namely, the acetate groups.

Further, the present study has shown that the xylan in a birch sulfate pulp can be divided into fractions with quite different 4-*O*-methyl-D-glucuronic acid content. Although the overall content of uronic acid in the xylan decreases during the cook, a considerable amount remains combined with the xylan in the pulp. When the temperature reaches the level (160–170°C.) where the rate of the alkaline hydrolysis of the uronic acid is significant, a large part of the alkali has already been consumed and therefore the loss of uronic acid from the xylan is not as great as would otherwise be expected.

As a xylan with 4 to 5% uronic acid content has a strong tendency to be adsorbed by the cellulose, and as uronic acid groups portect the xylan from being degraded by a peeling-off reaction, it is likely that complete removal of the uronic acid in not necessary for a firm stabilization of the xylan during a sulfate cook of birchwood.

## EXPERIMENTAL

*Paper Chromatography.* Paper: whatman no. 1. Solvents: ethyl acetate, acetic acid water 9:2:2; butanol, acetone, water, 4:5:1. Developer: aniline phthalate.

*Preparation of the Birch Holocellulose.* Chlorite holocellulose from acetone extracted wood meal of *Betula verrucosa* was prepared according to a slightly modified Wise method (*3*).

*Fractional Extraction of the Holocellulose.* Holocellulose (1 part) was extracted with alkali (10 parts) (5% KOH and 10% NaOH, respectively) for 5 hr. in a stainless steel autoclave. The air in the autoclave was replaced by nitrogen by alternative evacuations and nitrogen refillings. After acidification with acetic acid, the precipitate ($\beta$-fraction) was isolated by centrifugation in a Servall centrifuge (relative Centrifugal force = 27,000 *g.*). The $\gamma$-fraction was isolated by precipitation in alcohol, followed by centrifugation.

In another experiment the milled holocellulose (50 g., 40–60 mesh) was given a stepwise extraction (in the same autoclave) with sodium hydroxide (500 ml.) of the following concentrations 0.1, 0.5, 1, 2, 4, 5, and 10%. The filtrates were acidified with acetic acid and the $\beta$-fractions were isolated as above. These fractions were washed twice with water (these washings being combined with the first centrifugates), once with alcohol-water (4:1), three times with acetone and twice with ethyl ether. The $\gamma$-fractions were precipitated separately in alcohol, washed with alcohol-water (4:1), acetone, and ether. All fractions were dried *in vacuo* at 50°C.

*Preparation of the Sulfate Pulps.* The sulfate cooks were carried out in electrically heated 10-liter tumbling digesters. After cooking, the pulps were all washed with hot and cold water, screened on a laboratory screen with 0.30 mm. slotted plates, centrifuges, disintegrated, conditioned, and weighed.

The cooking conditions and some pulp characteristics are given in Table VI. The temperature was raised to 170°C. in 2.5 hr., and kept there for another 2 hr.

### *Isolation of Xylan from the Sulfate Pulps*

The pulps were deresinified with methylene chloride before the extractions.

Table VI

| Pulp | Active alkali as kg. NaOH/ton of wood | pH | Cooking yield, % | Screenings % of wood | Roe no. | TAPPI viscosity cp. |
|---|---|---|---|---|---|---|
| 1 | 170 | 10.55 | 54.1 | 7.5 | 5.0 | 267 |
| 2 | 200 | 11.05 | 51.8 | 0.2 | 2.8 | 97 |

| $R_{18}$, % | $R_{10}$, % | $\bar{R}$, % | Brightness, % G.E. | Pentosans, % | Resin, % |
|---|---|---|---|---|---|
| 89.2 | 85.8 | 87.5 | 22.6 | 23.1 | 1.55 |
| 92.0 | 89.1 | 90.6 | 28.6 | 24.0 | 1.31 |

*Extraction with Dimethyl Sulfoxide.* The pulp (50 g.) was extracted twice with dimethyl sulfoxide (1 l.) in a stainless steel autoclave. After each extraction, the pulp was washed with dimethyl sulfoxide (500 ml.). The xylan was precipitated in alcohol (3 times the volume of the extract) acidified with 2 to 3 ml. hydrogen chloride. The xylan fractions were combined and washed with acetone–petroleum ether (4:1) and ether and then dried *in vacuo* at 50°C.

*Extraction with Alkali.* The pulps were extracted in the same way as the holocellulose, described above, except that the 5%-KOH solution was replaced by a 5%-NaOH solution.

***Isolation of 4-O-Methyl-D-glucuronic Acid from the Sulfate Pulp Xylan***

Xylan (20 g.), isolated from the 5%-NaOH extract of pulp I, was dissolved in 72% sulfuric acid (68 ml.). After 15 min., the solution was diluted with water to 800 ml., heated for 3.5 hr. on a water bath, neutralized to pH 3.5 with a warm, saturated barium hydroxide solution, and then filtered through celite. The filtrate was treated with a cation exchange resin (Amberlite IR-120, $H^+$-form). The uronic acids were adsorbed on an anion exchange column (3.5 by 40 cm.) (Dowex 2, acetate form). The acids were then eluted using a linear gradient of water–2*N* acetic acid. Fractions (20 ml.) were collected using an automatic fraction collector. In this way the neutral sugars and 4-*O*-methyl-D-glucuronic acid were separated from the aldobiouronic acid, which was obtained in a chromatographically pure form.

The aldobiouronic acid was transformed into its methyl ester methyl glycoside derivative, part of which was reduced with lithium aluminum hydride. Hydrolysis of the reduced material gave xylose and 4-*O*-methylglucose as the sole products. Another part was converted to the crystalline tetraacetate according to Timell (*13*). The m.p. was 199–200°C. and $[\alpha]_D^{25} = 98°$ (*c*, 1.5 in chloroform). The infrared spectra showed characteristic adsorptions at the following wave numbers 2940, 1745, 1440, 1370, 1225, 1050, 965, 920, 885, 790, 740, and 655 $cm.^{-1}$.

***Xylan Adsorption Experiments (3)***

The cellulose material (cellulose linters and a sulfite high $\alpha$-cellulose pulp) (0.5 g.) was mixed with a solution (17 ml.), containing 5 g. xylan/100 ml. sodium hydroxide solution. Two different solutions containing 0.5 and 2% sodium hydroxide, respectively, were used. The samples were heated in autoclaves for 3 hr. at 170°C., after which the cellulose material was filtered and washed with warm water, cold water, acetone, and then dried *in vacuo* at 50°C. Before and after the cook, 5 ml. of the solution were titrated to pH 8 with 0.1*N* HCl in order to estimate the alkali consumption during the cook. The sugar composition of the pulps was analyzed by paper chromatography. Most of the adsorbed xylan (92%) could be removed by extraction with 10% sodium hydroxide. After the precipitation and purification, the xylan was analyzed for sugar composition and uronic acid content.

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# The Elasticity of Paper and Other Fibrous Sheets*


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**A theory is presented in order to describe the density, elasticity and dynamic viscoelasticity of paper and other fibrous sheets in terms of their structure. The network model on which the theory is based is almost identical with that employed by Nimura and Kido, but the mathematical treatments are different. On computing the number of segments in a unit cube, the effect of the thickness of constituent fibers is considered. This leads to excellent agreement between theoretical and experimental results. The density and the modulus of elasticity of a sheet are proportional, respectively, to those of the constituent fiber, and increase rapidly with the number of junctions. The correlation between the modulus and the density of the sheet depends markedly upon the effect of the thickness and the frequency probability of the inclination of segments to the plane of the sheet. The relaxation spectrum for one type of paper has been determined from the dynamic data and compared with those of cellulosic fibers. The former, corrected for the structural factor, shows no essential difference from the latter, showing that the most important assumption in the theory is valid.**


THE MECHANICAL properties of paper and other fibrous sheets are very important for their usefulness.


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*The theoretical part of this paper was published in *Japanese Tappi* 4. 233–238 (1957).

Among them, elasticity is a measure of resistance to deformation and is one of the fundamental properties in a different sense from various breaking strengths, which have rather practical meanings. Elasticity is also very important in practice. In general, the modulus of elasticity of papers is directly proportional to the flexural rigidity, and it is no exaggeration to say that the adaptability of the paper cone for loud speakers depends solely on its elastic properties. We can find another example in pulp sheets: the elasticity of dissolving pulp sheets can be an excellent measure of their quality (*1*).

Although many studies on the elasticity and viscoelasticity of fibrous sheets have been carried out hitherto, almost all of them are restricted only to phenomenological treatments, without considering the structure of the sheets. The mechanical properties of such sheets, of course, are directly connected with the nature and texture of the constituent fibers, namely with the structure of the sheets. Therefore, one cannot discuss the mechanical properties of the sheets separate from their structures. In this meaning, Hurley (*2*) as well as Nimura and Kido (*3*) tried to clarify the relation between the structure and elastic properties of paper by employing some models. Hurley's model, however, is too simple to explain the existing experimental results very well. On the other hand, Nimura and Kido obtained good results by employing a generalized model and solving very difficult problems. But, unfortunately, the effect of thickness of constituent fibers was neglected in their theory, and hence the results lack generality.

In the present paper, it has been intended to extend the above theory to derive some properties such as density, modulus of elasticity, and viscoelasticity as functions of structural factors from a network model and to explain more precisely the correlation between modulus of elasticity and density, which has been studied extensively by us (*4–6*). The network model employed here and the fundamental equations are almost the same as those of Nimura and Kido, but the effect of the thickness of constituent fibers has been taken into consideration in our calculations. The results obtained are more general and can explain experimental results more satisfactorily. The formulas for the viscoelasticity have also been derived. A further extension should be made to explain the breaking strengths by introducing slippage or breaking of junctions between fibers, but such an extension does not appear to be easy to perform.

## NOMENCLATURE

| | |
|---|---|
| $l$ | = length of segment, cm. |
| $\theta$ | = angle between segment and $Z$-axis. |
| $\varphi$ | = angle between project of segment and $X$-axis. |
| $F_0$ | = external force acting on segment, dynes. |
| $\xi_0$ | = deformation of segment, cm. |
| $E_0$ | = Young's modulus of segment or fiber, dynes/cm². |
| $A$ | = area of cross-section, cm². |
| $I$ | = moment of inertia of cross-section about axis perpendicular to plane of bending, cm⁴. |
| $N_x$, $N_y$, $N_z$ | = number of segments in $X$, $Y$, and $Z$-directions. |
| $F$ | = force acting on unit area of sheet, dynes. |
| $\xi$ | = deformation of unit cube of sheet, cm. |
| $E$ | = Young's modulus of sheet, dynes/cm². |
| $\rho$ | = density of sheet, g./cc. |
| $\rho_0$ | = density of segment or fiber, g./cc. |
| $E^*$ | = complex modulus of sheet, dynes/cm². |
| $E_0^*$ | = complex modulus of fiber, dynes/cm². |
| $E'$ | = storage modulus of sheet, dynes/cm². |
| $E_0'$ | = storage modulus of fiber, dynes/cm². |
| $E''$ | = loss modulus of sheet, dynes/cm². |
| $E_0''$ | = loss modulus of fiber, dynes/cm². |
| $\eta'$ | = dynamic viscosity of sheet, dynes·sec/cm². |
| $\eta_0'$ | = dynamic viscosity of fiber, dynes·sec/cm². |
| $l$, $m$, $n$ | = three components of direction cosines of normal to two segments. |
| $\bar{l}$, $\bar{m}$, $\bar{n}$ | = mean values of $l$, $m$, and $n$. |
| $r$ | = radius of circular cross-section of segment or fiber, cm. |
| $\beta$ | = coefficient which determine the effect of thickness of segments. |
| $H(\tau)$ | = relaxation spectrum, dynes/cm². |
| $\lambda(l)$, $\Theta(\theta)$, $(\Gamma\varphi)$, $\Phi(I)$, $\Psi(A)$, $\chi(r)$ | = frequency probabilities. |

## MODEL AND FUNDAMENTAL CALCULATIONS

As a model for fibrous sheets, a three-dimensional network structure as shown in Fig. 1 is employed, in which a number of thin fibers entangle with one another. It is supposed that the number of junctions is so large that an external force can be transmitted uniformly and that deformation is also uniform. Further assumptions for the model are: (1) a portion of a fiber between two adjacent junctions, referred to as a segment hereafter, is straight. This assumption may be satisfied sufficiently so far as numerous junctions exist. (2) The junctions are strong enough so that no slippage occurs at the junctions when the sheet deforms. This may be satisfied when a deformation is as small as that in dynamic tests, but it should be modified for larger deformations. (3) When the sheet is subjected to a uniform tension, each segment deforms without any change in its direction at junctions relative to coordinates fixed in space. In other words, no local rotation of fibers occurs, and a deformation of the sheet is due not to the rotation, but to the elastic elongation and bending of the segments.

Besides these assumptions, in order to allow the model to resemble the structures of actual sheets, a consideration will be taken into our calculations that fibers in the sheet do not orient uniformly, but lie preferentially in the plane of the sheet.

On the basis of the above model, we can calculate not only the density, but also the elastic properties of a sheet subjected to a uniform external force. We take out any one segment and fix its one end at the origin $O$ of the coordinates. Another end, $P$, of the segment is at $(l, \theta, \varphi)$ in polar coordinates, as shown in Fig. 2, and at this end an external force $F_0$ parallel to the $X$-axis acts on the segment. When the force $F_0$ is resolved into two components, one parallel and the other perpendicular to the segment, the displacement of $P$ in the direction of $X$, $\xi_0$, is given as the vector sum of two displacements $x_0$ and $y_0$ due to the two components of the force:

$$\xi_0 = (x_0 \sin\theta\cos\varphi + y_0\sqrt{1 - \sin^2\theta\cos^2\varphi}) = \frac{lF_0}{E_0}\left\{\frac{1}{A}\sin^2\theta\cos^2\varphi + \frac{l^2}{12I}(1 - \sin^2\theta\cos^2\varphi)\right\} \quad (1)$$

The first term on the right-hand side of this equation shows the elastic elongation of the segment, and the second the elastic bending. Similarly, we can get the displacement in the $YZ$-plane.

Now we take a unit cube of the sheet and assume the numbers of segments in the $X$, $Y$, and $Z$ directions to be

$N_x$, $N_y$, and $N_z$, respectively. Then, the following relations hold for the $X$, $Y$, and $Z$ directions, respectively:

$$1 = N_y^z \int_0^\infty \lambda(l) \int_0^{\pi/2} \Gamma(\varphi) \int_0^{\pi/2} \Theta(\theta) l \sin\theta \begin{pmatrix}\cos\\ \sin\end{pmatrix} \varphi \, d\theta d\varphi dl \tag{2}$$

and

$$1 = N_z \int_0^\infty \lambda(l) \int_0^{\pi/2} l \cos\theta \, \Theta(\theta) \, d\theta dl \tag{3}$$

In these equations, $\lambda(l)$, $\Theta(\theta)$, and $\Gamma(\varphi)$ are the frequency probabilities to represent the distribution of $l$, $\theta$, and $\varphi$, respectively, and the effect of the thickness of the segments is not taken into consideration.

Now, we can consider that the unit cube of the sheet is formed by $N_yN_z$ chains consisting of $N_x$ segments, and hence the displacement between two ends of a chain in the $X$-direction, $\xi$, is given by the following equation:

$$\begin{aligned}\xi &= N_x \int_0^{\pi/2} \Theta(\theta) \int_0^\infty \lambda(l) \int_0^\infty \Phi(I) \int_0^\infty \Psi(A) \xi_0 dA dI dl d\theta \\ &= \frac{N_x}{N_y N_z} \frac{F}{E_0} \int_0^{\pi/2} \Theta(\theta) \int_0^\infty \lambda(l) \int_0^\infty \Phi(I) \int_0^\infty \Psi(A) l \\ &\times \left\{ \frac{1}{A} \sin^2\theta \cos^2\varphi + \frac{l^2}{12I} (1 - \sin^2\theta \cos^2\varphi) \right\} dA dI dl d\theta \end{aligned} \tag{4}$$

where $\Phi(I)$, $\Psi(A)$ are the frequency probabilities with respect to $I$ and $A$, and $F$ is a force per unit area. Since the displacement of the whole unit cube is equal to the above $\xi$, the Young's modulus of the sheet can be given by:

$$E = \frac{F}{\xi} = \frac{N_y N_z E_0}{N_x \iiint \Theta(\theta)\lambda(l)\Phi(I)\Psi(A) l \times \left\{ \frac{1}{A} \sin^2\theta \cos^2\varphi + \frac{l^2}{12I} (1 - \sin^2\theta \cos^2\varphi) \right\} dA dI dl d\theta} \tag{5}$$

When we calculate the displacements in the $Y$ and $Z$ directions in a similar manner and obtain those ratios to $\xi$ in equation (4), Poisson's ratios can be obtained.

The apparent density of the sheet, on the other hand, is a product of the total volume of segments in a unit volume and the density of the fiber itself, $\rho_0$, it can be given by:

$$\rho = N_x N_y N_z \int_0^\infty \lambda(l) \int_0^\infty \Psi(A) A l \rho_0 dA dl \tag{6}$$

The above calculations were carried out by assuming the sheet to be an elastic body. But, if the sheet is viscoelastic and the force $F$ can be given as a sum of elastic and viscous components, an equation similar to equation (5) holds for the complex modulus of elasticity $E^*$ of the sheet. To obtain this equation one may replace $E_0$ in equations (1) and (4) by the complex modulus of fiber itself, $E_0^*$, and the result is as follows:

$$E^* = \frac{N_y N_z E_0^*}{N_x \iiint \Theta(\theta)\lambda(l)\Phi(I)\Psi(A) l \times \left\{ \frac{1}{A} \sin^2\theta \cos^2\varphi + \frac{l^2}{12I} (1 - \sin^2\theta \cos^2\varphi) \right\} dA dI dl d\theta} \tag{7}$$

*Fig. 1. Three-dimensional network as a model of sheets*

Since $E^*$ and $E_0^*$ can be defined by the equations, respectively,

$$E^* = E' + iE'' = E' + i\omega\eta' \tag{8a}$$

$$E_0^* = E_0 + iE_0'' = E_0' + i\omega\eta_0' \tag{8b}$$

the real (storage modulus $E'$) and imaginary (loss modulus $E''$) parts of $E^*$, and the dynamic viscosity $\eta'$ can be given by equations of the same form as equation (7), in which $E^*$ and $E_0^*$ are replaced by $E'$ and $E_0'$, $E''$ and $E_0''$, and $\eta'$ and $\eta_0'$, respectively.

## CALCULATION OF $N_x$, $N_y$ AND $N_z$ AND FORMS OF THE FREQUENCY PROBABILITIES

To obtain useful results from the foregoing equations, further calculations must be carried out. Beforehand, true numbers of segments in unit cube of the sheet

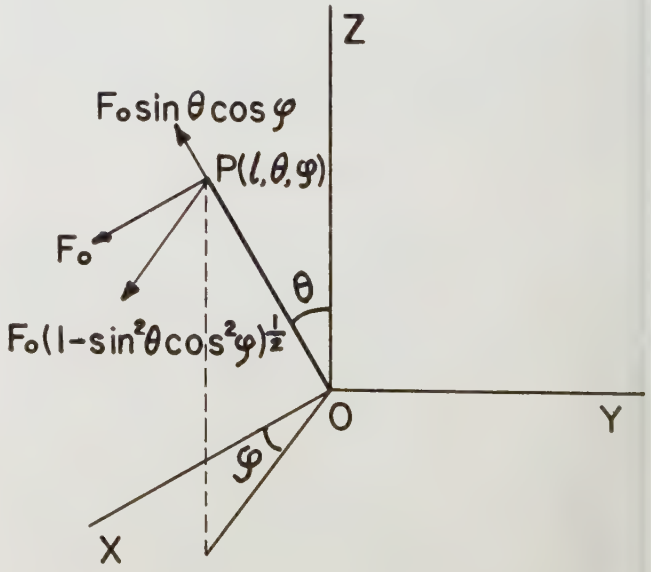


*Fig. 2. Polar coordinates of a segment in sheet*

$N_x$, $N_y$, and $N_z$, including the effect of the thickness, should be determined. They cannot be determined accurately only from the length and direction of the segments, but the effect of their thickness must also be considered. So far as we neglect the effect of the thickness, equations (2) and (3) hold for $N_x$, $N_y$, and $N_z$. But the thickness of the segments must decrease the number of segments in each direction, because it makes some contribution to the length of the segment. And, so far as the length and arrangement of the segments are the same, the thicker the constituent segments, the smaller the number of segments in each direction of the unit cube.

To determine the contribution of the thickness to the length, we should calculate the dimensions of three projections of the cross-section to the $X$, $Y$ and $Z$-planes. For simplicity, we suppose that a segment having direction cosines ($\sin\theta_0\cos\varphi$, $\sin\theta_0 \sin\varphi$, $\cos\theta_0$) intersect with a given segment of direction cosines ($\sin\theta_0$, 0, $\cos\theta_0$) at the origin. Then the $Z$-component $n$ of the direction cosine of the normal to these two segments at the intersection is given by:

$$n = \frac{\tan\theta_0}{\left\{\frac{1}{\cos^2\theta_0} + \left(\frac{1-\cos\varphi}{1+\cos\varphi}\right)\right\}^{1/2}} \qquad (9)$$

When the distribution of segments with respect to $\varphi$ is uniform, the average value of $n$ is given as follows:

$$\bar{n} = \int_0^{\pi} \Phi(\varphi) n d\varphi = \frac{1}{\pi}\sin^{-1}(2\overline{\sin^2\theta_0} - 1) + \frac{1}{2} \qquad (9a)$$

where,

$$\Phi(\varphi) = \frac{1}{\pi}, \text{ and } \overline{\sin^2\theta_0} = \int_0^{\pi/2} \Theta(\theta)\sin^2\theta d\vartheta$$

On the contrary, the average value of the $X$ and $Y$-components, $\bar{l}$ and $\bar{m}$, is given by the following ellipsoidal integral:

$$\bar{l} = \bar{m} = \frac{2}{\pi}\int_0^{\pi} \Phi(\varphi)\sqrt{1-n^2}\, d\varphi = \frac{2}{\pi^2}\int_0^{\pi} \frac{\overline{\cos\theta_0}}{\sqrt{1-\sin^2\vartheta_0\sin^2\frac{\varphi}{2}}}\, d\varphi \qquad (10)$$

where

$$\overline{\cos\theta_0} = \int_0^{\pi/2} \Theta(\vartheta)\cos\theta\, d\theta$$

Therefore, when all segments have circular cross-sections, and the frequency probability of the radius $r$ is denoted by $\chi(r)$, the contributions of the thickness to the $X$, $Y$, and $Z$-directions are given by:

$$\bar{l}\int_0^{\infty} 2r\chi(r)dr,\ \bar{m}\int_0^{\infty} 2r\chi(r)dr, \text{ and } \bar{n}\int_0^{\infty} 2r\chi(r)dr$$

These contributions, however, need not always be taken into consideration. For example, when both segments belong to the same fiber, they need not be considered. And in a special or idealized case in which all fibers lie parallel to the plane of the sheet, the number of segments in the direction perpendicular to this plane is determined only by the radius of fibers. Consequently, it is reasonable to consider that the above contributions must be multiplied by a factor $\beta$ equal to or less than unity. So far as the number of junctions is large enough and we can always make a choice of segment intersecting with a given segment only on its half circumference in plus direction, the lower limit of $\beta$ is considered statistically to be $1/4$. When the number of junctions is not so large, contrary to our assumption, $\beta$ may be greater than $1/4$. The results obtained by Nimura and Kido correspond to the case in which $\beta = 0$, but, as shown below, the values of the modulus of elasticity and density depend largely upon the value of $\beta$.

Now, employing the above results for $\bar{l}$, $\bar{m}$, and $\bar{n}$, the numbers of segments in the $X$, $Y$, and $Z$-directions are given as follows:

$$1 = N_x\left[\int_0^{\infty}\lambda(l)\int_0^{\pi/2}\Gamma(\varphi)\int_0^{\pi/2}\Theta(\theta) l\sin\theta\cos\varphi\, d\theta d\varphi dl + \beta\bar{l}\int_0^{\infty} 2r\chi(r)dr\right] \qquad (11)$$

$$1 = N_y\left[\int_0^{\infty}\lambda(l)\int_0^{\pi/2}\Gamma(\varphi)\int_0^{\pi/2}\Theta(\theta) l\sin\theta\sin\varphi\, d\theta d\varphi dl + \beta\bar{m}\int_0^{\infty} 2r\chi(r)dr\right] \qquad (12)$$

$$1 = N_z\left[\int_9^{\infty}\lambda(l)\int_0^{\pi/2}\Theta(\theta) l\cos\theta\, d\theta\, dl + \beta\bar{n}\int_0^{\infty} 2r\chi(r)dr\right] \qquad (13)$$

The next step is to give analytical forms to the frequency probabilities in equations (5), (6) and (11), (12), and (13). To simplify the later calculations, it may be advisable to choose the simplest forms for the frequency probabilities unless they neglect the important part of the problem.

At first, we assume the frequency probability of the length $l$ to be $\lambda(l) = \delta(l - l_0)$, where $\delta(l)$ is the delta function of Dirac. This means that all segments have a uniform length $l_0$. Similarly, all segments are assumed to have circular cross-sections of the same radius $r_0$. Then, $A = \pi r_0^2$, $I = \pi r_0^4/4$, and the frequency probabilities $\chi(r)$, $\Psi(A)$, and $\Phi(I)$ can be represented by delta functions like $\delta(l)$. The cross-sections of real fibers, of course, are not circular but approximately ellipsoidal. However, any choice other than a circular cross-section does not change the essential parts of our problem.

The frequency probability of $\varphi$, the angle between the project of the segment and the $X$-axis, is also assumed to be $\Gamma(\varphi) = 2/\pi$. This means that the distribution of segments with respect to $\varphi$ is uniform, and that segments have no particular orientation in the $XY$-plane. When we consider the orientation of fibers in the plane of the sheet, we may modify the form of the function by other suitable ones.

Next, the frequency probability of $\theta$, the angle between the segment and the $Z$-axis, is considered as follows. As described above, most segments in the sheet lie almost parallel to the plane of the sheet or the $XY$-plane, and segments making larger angles with this plane should be very few. Therefore, the following functions will be very suitable for the frequency probability of $\theta$.

$$\Theta(\theta) = \frac{\alpha(\alpha-2)\cdots 2}{(\alpha-1)(\alpha-3)\cdots 1}\cdot\frac{2}{\pi}\sin^{\alpha}\theta, \qquad \alpha = \text{even}$$

or

$$\Theta(\theta) = \frac{\alpha(\alpha-2)\cdots 1}{(\alpha-1)(\alpha-3)\cdots 2}\sin^{\alpha}\theta, \qquad \alpha = \text{odd}$$

For the limiting case where all segments lie parallel

the $XY$-plane, the frequency probability is given by the delta function again. Contrary to this, when the distribution is uniform, the probability becomes $\Theta(\theta) = \sin\theta$. Employing these frequency probabilities as well as $N_x$, $N_y$, and $N_z$ given above, we can calculate the values of the density and the elastic properties of the sheet. Several examples will be shown below.

## EXAMPLES OF PRACTICAL CALCULATIONS

Giving three different forms to $\Theta(\theta)$, the modulus of elasticity and the density have been calculated. The results will be compared with the experimental data and will be discussed.

*Case 1:* $\Theta(\theta) = \delta(\theta - \pi/2)$

This is an idealized case in which all segments lie in the plane of the sheet. $N_x$, $N_y$, and $N_z$ reduce, respectively, to $N_x = N_y = \pi/2l_0$, $N_z = 1/2r_0$

Consequently, Young's modulus $E$ and the density $\rho$ of the sheet can be given as:

$$E = \frac{\pi E_0}{\left(\frac{l_0}{r_0}\right)\left\{\frac{1}{3}\left(\frac{l_0}{r_0}\right)^2 + 1\right\}} \tag{14}$$

$$\rho = \frac{\pi^3}{8}\left(\frac{r_0}{l_0}\right)\rho_0 \tag{15}$$

Equation (14) can also be written as follows:

$$E = \frac{\pi E_0}{\frac{\pi^3\rho_0}{8\rho}\left(\frac{1}{3}\frac{\pi^6\rho_0^2}{64\rho^2} + 1\right)} \doteqdot \frac{3\cdot 8^3\cdot\rho^3}{\pi^8\rho_0^3}E_0 \tag{16}$$

As seen from these results, $E$ and $\rho$ of the sheet are proportional to $E_0$ and $\rho_0$ of the fibers, respectively, and increase with increasing $l_0/r_0$. Namely, $E$ and $\rho$ increase as the segments become shorter at constant thickness; i.e., they increase as the number of junctions between fibers increases. This special feature can also be seen in the other cases shown below, and is considered to be quite reasonable.

Furthermore, it is clear from the above equations that $E$ is almost proportional to the cube of $l_0/r_0$, and $\rho$ is inversely proportional to $l_0/r_0$. Hence, Young's modulus is approximately proportional to the cube of the density. Equation (16) shows this fact.

In the above treatments, the length and thickness of the segments were assumed to be uniform, but when they show wide distributions, the modulus decreases and the density increases. Namely, the modulus for a given density becomes smaller.

*Case 2:* $\Theta(\theta) = \dfrac{6\cdot 4\cdot 4\sin^6\theta}{5\cdot 3\cdot\pi}$

As described above, Case 1 is the idealized one and cannot be realized for actual sheets. Therefore, we employ here the above form of distribution. After some simple calculations, we obtain:

$$\overline{\sin^2\theta_0} = \frac{6\cdot 4\cdot 4}{5\cdot 3\cdot\pi}\int_0^{\pi/2}\sin^8\theta\,d\theta = 0.718$$

$$\overline{\cos\theta_0} = \frac{6\cdot 4\cdot 4}{7\cdot 5\cdot 3\pi} = 0.291$$

$$\bar{n} = \frac{1}{\pi}\sin^{-1}\frac{3}{4} + \frac{1}{2} = 0.770$$

$$\bar{l} = \bar{m} = \frac{2}{\pi^2}\,\overline{\cos\theta_0}\int_0^{\pi}\frac{d\varphi}{\sqrt{1 - \overline{\sin^2\theta_0}\sin^2\frac{\varphi}{2}}}$$

$$= \frac{2}{\pi^2}\times 4.946\,\overline{\cos\theta_0}$$

$$= 0.292$$

Hence,

$$N_z = 1/(0.291l_0 + 1.584\,\beta r_0)$$

$$N_x = N_y = 1/(0.593l_0 + 0.584\,\beta r_0)$$

Consequently, $E$ and $\rho$ are given as follows:

$$E = \frac{48\pi E_0}{\left(\frac{l_0}{r_0}\right)\left\{0.291\left(\frac{l_0}{r_0}\right) + 1.54\beta\right\}\left\{9\left(\frac{l_0}{r_0}\right)^2 + 7\right\}} \tag{17}$$

$$\rho = \left\{\frac{1}{0.593\left(\frac{l_0}{r_0}\right) + 0.584\beta}\right\}^2\left\{\frac{1}{0.291\left(\frac{l_0}{r_0}\right) + 1.54\beta}\right\} \times \pi\left(\frac{l_0}{r_0}\right)\rho_0 \tag{18}$$

Similarly to Case 1, $E$ and $\rho$ are proportional to $E_0$ and $\rho_0$, respectively, and increase with decreasing value of $l_0/r_0$, or with increasing number of junctions. But, in this case $E$ and $\rho$ depend not only on $l_0/r_0$, but also on $\beta$.

*Case 3:* $\Theta(\theta) = \sin\theta$

This is the case of uniform distribution of segments with respect to $\theta$. Here,

$$\overline{\sin^2\theta_0} = \int_0^{\pi/2}\sin^3\theta\,d\theta = \frac{2}{3}$$

$$\overline{\cos\theta_0} = \int_0^{\pi/2}\cos\theta\sin\theta\,d\vartheta = \frac{1}{2}$$

$$\bar{n} = \frac{1}{\pi}\sin^{-1}\frac{1}{3} + \frac{1}{2} = 0.608$$

$$\bar{l} = \bar{m} = \frac{2}{\pi^2}\cdot\frac{1}{2}\int_0^{\pi}\frac{d\varphi}{\sqrt{1 - \frac{2}{3}\sin^2\frac{\varphi}{2}}} = 0.411$$

and hence,

$$N_z = \frac{2}{l_0 + 2.43\,\beta r_0}$$

$$N_x = N_y = \frac{2}{l_0 + 1.64\beta r_0}$$

Consequently, $E$ and $\rho$ are given by:

$$E = \frac{6\pi E_0}{\left(\frac{l_0}{r_0}\right)\left\{\left(\frac{l_0}{r_0}\right) + 2.43\beta\right\}\left\{\frac{2}{3}\left(\frac{l_0}{r_0}\right)^2 + 1\right\}} \tag{19}$$

$$\rho = \frac{8\pi}{\left\{\left(\frac{l_0}{r_0}\right) + 1.64\beta\right\}^2\left\{\left(\frac{l_0}{r_0}\right) + 2.43\beta\right\}}\left(\frac{l_0}{r_0}\right)\rho_0 \tag{20}$$

The special features of $E$ and $\rho$ in this case are quite the same as in the former cases.

The values of $E$ and $\rho$ in the above three cases can be evaluated provided that the values of $l_0/r_0$, $\beta$, $E_0$, and $\rho_0$ are known. As an example, the values of $E$ and $\rho$ obtained by assuming $E_0 = 3 \times 10^{11}$ dynes per sq. cm. and $\rho_0 = 1.5$ g. per cc. are shown below.

Figure 3 shows a typical example of Young's modulus ($E$) and density ($\rho$) plotted against $l_0/r_0$ when $\beta = 1$. As seen from this figure, $E$ and $\rho$ increase with decreasing $l_0/r_0$ in all cases, but the slopes are slightly different from one another, depending on the distribution with respect to $\theta$. Furthermore, the sharper the distribution, or the more parallel the segments lie to the plane of the sheet, the higher is the modulus and

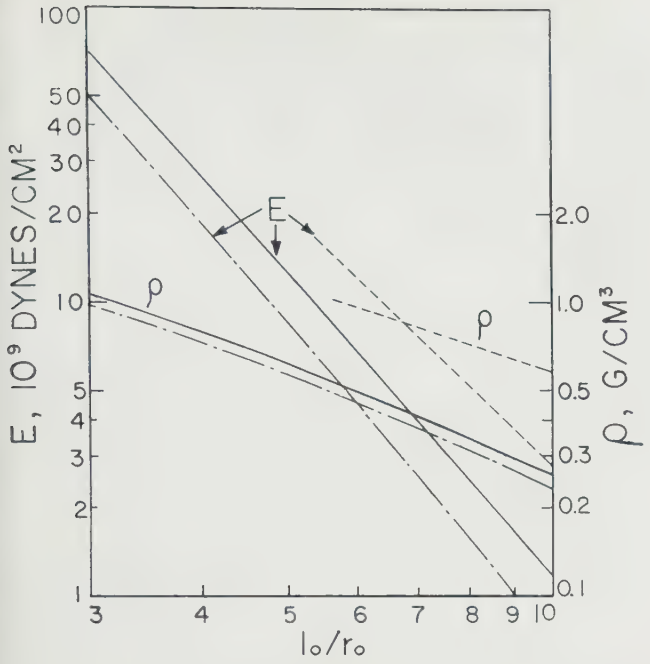


*Fig. 3. Young's modulus, E, and density, ρ, plotted against $l_0/r_0$ when $\beta = 1$*

— — — Case 1
——— Case 2
·—·—· Case 3

the density for a given value of $l_0/r_0$. The changes of $E$ and $\rho$ with respect to $l_0/r_0$ also depend markedly on the value of $\beta$, though it is not shown in this figure.

In Fig. 4 the modulus in Fig. 3 is plotted logarithmically as a function of the density for the three cases. For Case 2 and 3, the elasticity corresponding to

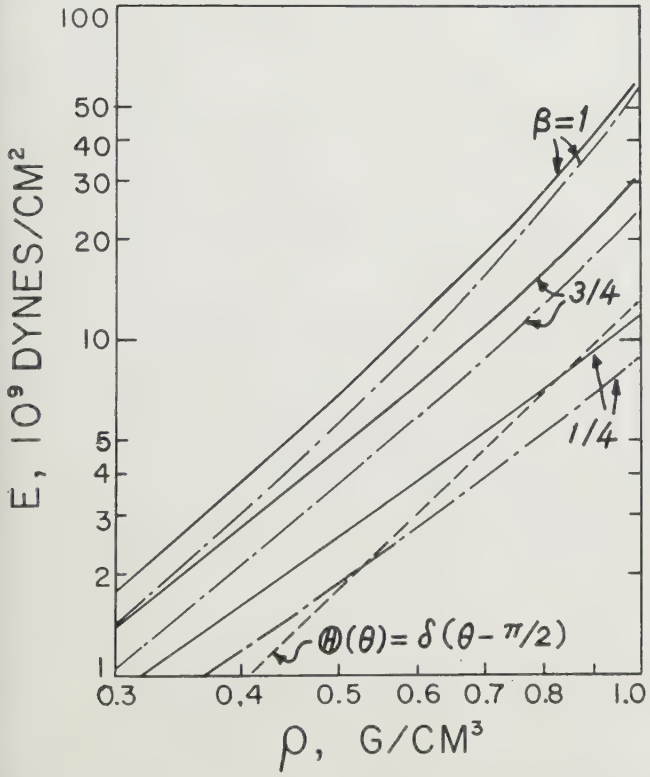


*Fig. 4. Correlation between Young's modulus, E, and density, ρ, in Cases 1–3*

— — — Case 1
——— Case 2
·—·—· Case 3

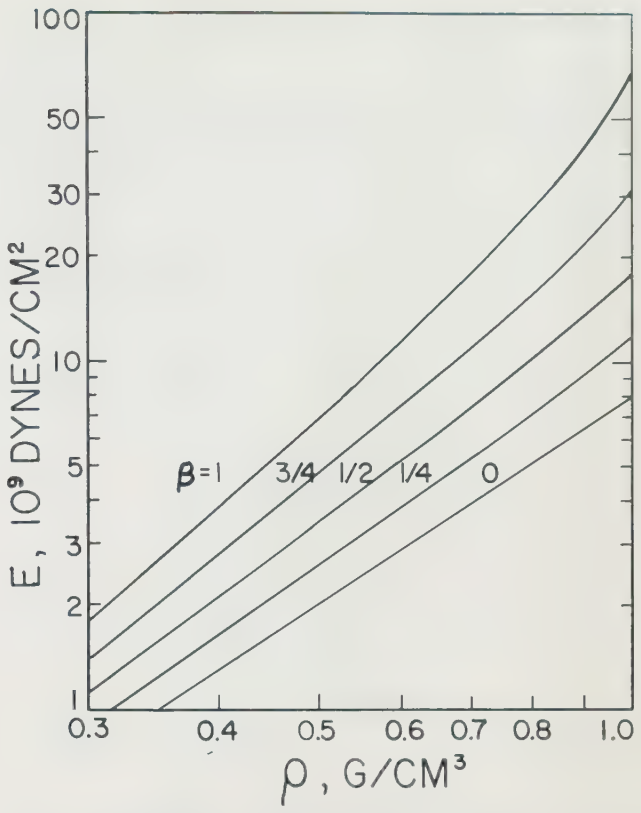


*Fig. 5. Dependency of the correlation between Young's modulus, E, and density, ρ, upon β*

$\beta = 3/4$ and $\beta = 1/4$ are also given. It is seen from the figure that log $E$–log $\rho$ curves for smaller values of $\beta$ are almost straight, and their slopes are slightly greater than 2. Only in the limiting case of $\beta = 0$ as treated by Nimura and Kido is the slope just equal to 2. Increasing the value of $\beta$, the curves bend upwards more and more, especially at higher densities. But, unless the range of density is very wide, the curves can be approximated by straight lines fairly well.

Figure 5 shows the influence of $\beta$ on the correlation between the elasticity and the density in more detail.

## DISCUSSION

It is very interesting to compare the results of this theory with the experimental data. In previous publications (*4–6*), the correlation between Young's modulus and the density for various papers and pulp sheets were studied by us very extensively. In the case of the usual papers, it has been shown statistically that the dynamic modulus plotted logarithmically against the logarithm of the density shows a straight

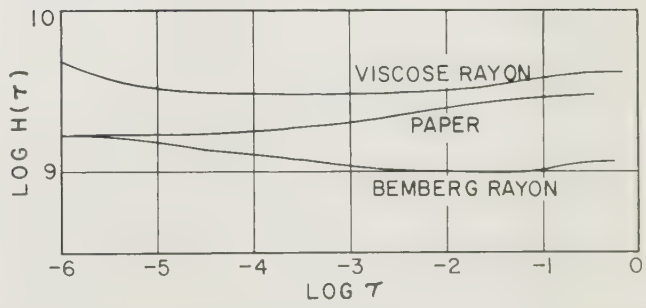


*Fig. 6. Relaxation spectrum of paper as compared with those of viscose and Bemberg rayons*

line of slope about 2. This corresponds to the theoretical result derived above for the case of smaller value of $\beta$. In the case of pulp sheets, on the other hand, it has been found that log-log plots of the modulus against the density for a group of sheets which differ only in their densities from one another show a straight-line behavior. Namely, the following relation between the modulus and the density holds for the group: $\log(E \cdot 10^{-8}) = k + n \log (10\ \rho)$, where $k$ is the intercept at log $(10\ \rho) = 0.3$, and $n$ the slope. Of these two constants, the former depends largely on the nature of the original stock and on the sheet-making conditions, especially press and drying conditions. The slope $n$, on the other hand, does not vary so widely, and ranges between 2 and 3 for substantially whole samples. In Table I, the previous results of $n$ and $k$ for various groups of sheets are reproduced *en bloc*. Values larger than 2 cannot be explained by the former theory, in which the effect of the thickness is neglected. The densities of the pulp sheets studied by us were very low as compared with those of usual papers. This means that the junctions between constituent fibers are not as numerous. Consequently, the value of $\beta$ is larger than its limiting value of $^1/_4$, and this is the reason why the log-log plots of the moduli against the density have a slope larger than 2.

**Table I. Applicability of the Equation log $(E \cdot 10^{-8}) = k + \log (10\rho)$, for Various Groups of Pulp Sheets**
$k$ is the intercept at log $(10\rho) = 0.3$

| *Species* | *Wood* | *Stock* | *Press* | k | n | *E, dynes/cm.² for ρ = 0.7* |
|---|---|---|---|---|---|---|
| C | Pine | Original | Wet press | 0.0665 | 2.27 | $9.66 \times 10^9$ |
| | | | Press after air-drying | −0.253 | 2.26 | 4.54 |
| | | Screened | Wet prrss | −0.428 | 2.58 | 5.65 |
| | | | Prcss after air-drying | −0.709 | 2.44 | 2.26 |
| A | Spruce and fir | Original | Wet press | −0.0142 | 2.14 | 6.23 |
| | | | Press after air-drying | −0.219 | 2.00 | 2.96 |
| | | Screened | Wet press | −0.626 | 2.76 | 5.09 |
| | | Purified with alkali | Wet press | −0.300 | 2.44 | 5.73 |
| F | Pine (American origin) | Original | Wet press | −0.593 | 2.52 | 3.44 |
| B | Pine | Subjected to seasoning for | | | | |
| | | 0 days | | | 2.83 | |
| | | 40 " | | | 2.83 | |
| | | 80 " | | | 2.78 | |
| | | Commercial product | | | 2.20 | |

Thus, the theory can derive the fundamental properties of fibrous sheets as functions of structural factors, and explain the existing experimental data very well. The coincidence between both the theoretical and experimental results is good evidence that the theory treats the essentials of the problem.

One of the most important assumptions in this theory is that the junctions between segments are strong enough to prevent any slippage or rotation of the segments at those points. This assumption appears to be satisfied very well, at least in the dynamic test. Equation (7) and the related equations show that the dynamic properties of the sheet can be represented as a product of the corresponding property of the constituent fibers themselves and the structural factors. Therefore, for instance, Young's modulus of the sheet is decreased by these structural factors from that of constituent fibers. The same holds true for other properties. If the above assumption is true, the dynamic properties of the sheet, corrected for structural factors, should be equal to those of the constituent fibers. To verify this, we determined the relaxation spectrum for one type of Japanese paper, and the result has been compared with the existing spectra of other cellulosic fibers because, unfortunately, the dynamic data for the wooden fibers in pulp or paper have not been available.

The paper was cut into a long strip with a width of 5 mm. in the machine direction and subjected to the dynamic tests at 25°C. and 65% R.H. by employing four methods, according to the frequency range. In the frequency range 0.2 to 10 c.p.s., the free-damping oscillation method was employed; from 50 to 220 c.p.s., the stretch-vibrometer similar to that of Lyons (*7*); from 1 to 5 kc., the longitudinal-wave resonance, and in higher range, the longitudinal-wave transmission originating from Nolle (*8*) were used, respectively. The details of the apparatus and measuring techniques have been described elsewhere (*9*). The relaxation spectrum $H(\tau)$ was calculated from the value of the loss modulus by using the so-called zero-order approximation method, and was corrected for the square of the density 0.43 (structural factor). The relaxation spectrum thus obtained versus relaxation time is shown in Fig. 6, with those of viscose and Bemberg rayons obtained by Fujino, Kawai and Horino (*7*). Three spectra show different slopes and intensities over the whole range, but the differences are unimportant and are rather slight as compared with the great differences in natures and structures of these samples. If there occurred any slippage between fibers at junctions in the paper, the spectrum of the paper would show some rapid decrease probably at longer times. But we cannot observe such an essential change in the spectrum.

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# Rheological Models and Laminar Shear Flow of Fiber Suspensions*

GEORGE BUGLIARELLO and JAMES W. DAILY

**The mechanics of the laminar and transition regimes of flow of fiber suspensions under shear are discussed. Analogies with rheological models of non-Newtonian fluids are used as frameworks for analyzing the actual behavior of suspensions as presented in a previous article (*1*). Generalized correlation of flow resistance data is considered. It is observed that laminar flow of fiber suspensions represents a particular type of non-Newtonian behavior due to a macroscopic suspended phase forming elastic networks through mechanical entanglement; common rheological models do not account for nonhomogeneity and do not describe fiber flows accurately; viscoelastic effects are present but stress-relaxation or stiffening was not evidenced; an amplified clear water annulus model with a Couette flow annulus agrees with the present results; transition to turbulence depends on fiber type and concentration and a criterion based on laminar sublayer thickness is suggested; Metzner type generalized flow resistance correlations depend on variable, and not easily obtained, parameters and are of limited usefulness.**

## 1. INTRODUCTION

THE TAPPI sponsored MIT experiments on the hydrodynamic characteristics of dilute fiber suspensions have provided a systematic set of information for a variety of fiber types and concentrations under a range of flow conditions. As described in a previous article (*1*), experiments in circular tubes included flow resistance over the laminar, transition, and turbulent regimes and velocity profiles and turbulence measurements in the turbulent regime. Flow resistance data were given and a comparison made for all the suspensions tested. The effects of fibers on velocity profiles were correlated with their effects on flow resistance. The present article will discuss the mechanics of the laminar and transition regimes.

Fiber suspensions are non-Newtonian in their behavior, suggesting the possibility of analogies with some of the rheological models of non-Newtonian fluids. Consequently it will be useful to present the common theoretical rheological classifications and use these as a framework for analyzing the actual behavior of suspensions as presented in the above mentioned article.

### NOMENCLATURE

| | |
|---|---|
| $c$ | = subscript denoting critical conditions |
| $C$ | = concentration, % (weight) also a constant in equation (20) |
| $d$ | = peripheral annulus thickness, ft. |
| $D$ | = tube diameter, ft. |
| $E$ | = modulus of elasticity, p.s.i. |
| $f$ | = friction factor $\{ = [\Delta P]/[\gamma(L/D)(V^2/2g)]$ |
| $f_c$ | = value of the friction factor at the end of the laminar regime |
| $g$ | = gravitational acceleration, 32.2 ft./sec.$^2$ |
| $\Delta H$ | = head loss, feet of water |
| $K,K'$ | = rheological parameters defined by equations (3) and (4) |
| $L$ | = lengths of pipe over which a given pressure drop $\Delta P$ occurs, ft.; also fiber length, mm. |
| $n,n'$ | = rheological parameters defined by equations (3) and (4) |
| $\Delta P$ | = pressure drop, lb./ft.$^2$ |
| $Q$ | = discharge, ft.$^3$/sec. |
| $r$ | = radial distance, ft. or in. |
| $R$ | = pipe radius, ft. or in. |
| $R_f$ | = radius of flat velocity region in velocity diagrams, ft. or in. |
| $R_p$ | = possible plug radius, ft. or in. |
| $\mathsf{R}$ | = Reynolds number in general, Newtonian Reynolds number for flow in circular pipe $(= VD/\nu)$ |
| $\mathsf{R}_d$ | = annulus Reynolds number $(= Vd/\nu)$ |
| $\mathsf{R}_L'$ | = generalized Reynolds number correlating $f$ for the laminar range |
| $t$ | = time, sec. |
| $t_D$ | = motion-decay time, sec. |
| $u$ | = local mean velocity, f.p.s. |
| $U_M$ | = mean velocity at the centerline, f.p.s. |
| $U_p$ | = plug velocity, f.p.s. |
| $V$ | = average bulk velocity (= discharge/area of cross section), f.p.s. |
| $w$ | = subscript denoting conditions at the wall |
| $W$ | = fiber width, $\mu$ |
| $x$ | = distance in the axial direction (mean flow direction), ft. |
| $y$ | = distance from the wall, ft. |
| $\delta'$ | = laminar sublayer thickness, ft. |
| $\gamma$ | = specific weight, lb./ft.$^3$ |
| $\mu$ | = molecular dynamic viscosity for Newtonian fluids $[= \tau/(du/dy)]$, lb.-sec./ft.$^2$ |
| $\mu_s$ | = slope viscosity, lb.-sec./ft.$^2$ |
| $\mu_{effective}$ | = effective viscosity for non-Newtonian fluids, lb.-sec./ft.$^2$ |
| $\nu$ | = Newtonian molecular kinematic viscosity $(= \mu/\rho)$, ft./sec. |
| $\rho$ | = fluid density, mass (slugs)/ft.$^3$ |
| $\tau$ | = local shear stress, lb./ft.$^2$ |
| $\tau_s$ | = shear stress at the surface of the plug, lb./ft.$^2$ |
| $\tau_w$ | = wall shear stress, lb./ft.$^2$ |
| $\tau_y$ | = yield shear stress, lb./ft.$^2$ |

## 2. BRIEF SURVEY OF RHEOLOGICAL CLASSIFICATIONS

Rheology studies the relationship between shear stresses and deformation. Fluid systems (i.e., fluids, fluid mixtures or fluid-solid mixtures) can be broadly classified for engineering purposes in various categories. A first classification of engineering importance is according to whether their shear stress–strain rate (deformation rate) relationship varies with time or not: time-dependent and time-independent fluids.

A second classification is according to whether the shear stresses increase progressively faster or progressively slower than the strain rates, i.e., according to whether the flow curves (shear-stress versus rate-of-strain curves) are concave or convex toward the rate of strain axis (Fig. 1). In the first case (which could be called stress "stiffening") the fluid is commonly referred to as a "rheopectic" if it is time-dependent,

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and as a "dilatant" if it is time-independent.† In the second case (stress "relaxing") the time-dependent behavior is labeled "thixotropic" and the time-independent is labeled "pseudoplastic."

In the border case in which the shear stress varies exactly in proportion to the rate of strain the fluid is called Newtonian, and is represented in the flow diagram by a straight line issuing from the origin.

Finally there may be some fluids which require the application of a shear stress of finite magnitude (yield stress) before flow can take place. The simplest case is the so-called Bingham plastic in which there is direct proportionality between stresses and rates of strain, after the initial yield stress has been overcome; its flow diagram is a straight line issuing from the point of the shear stress axis at which $\tau = \tau_y$.

It is important to stress at this point that the above classifications are based on the macroscopic phenomenology of fluid systems, as it becomes manifest through viscometer studies, and they do not always adequately reflect the intimate molecular characteristics of the fluid. It is quite possible that a consideration of the latter would lead to a physically more meaningful, generalized flow relationship, of which the various modes of behavior cited previously would be only particular cases, caused by the prevalence of some factors over others. Thus it has been suggested that all rheological behaviors are actually time-dependent, the so-called time-independent behavior being one in which stress adjustments of the fluid structure occur in such a short time as to defy observation.

Fiber suspensions, characterized by the presence of a solid phase of macroscopic dimensions enormously greater than the molecular dimensions of the suspending phase, probably would not benefit from a generalized approach based on molecular phenomena—except perhaps for its value as an analogy. Consequently, the following exposition will be confined only to the principal macroscopic modes of behavior.

The several types of behavior can be described to various degrees of accuracy by mathematical models. Historically the Newtonian behavior, which is the simplest and describes the most common fluids, particularly air and water, was the first to be described and represents the basis for the traditional mathematical theories for flow. Only more recently have the other types of behavior which are characteristic of an extremely wide range of fluids, from blood to volcanic magmas, attracted scientific interest and mathematical description. The amount of pertinent literature is accordingly very limited, but growing fast.

For purposes of discussion we will consider what have become the common elementary models for time-independent fluids. We should keep in mind that these models all inherently assume homogeneous fluids such that the mechanism of shear transmission is the same throughout fluid filled space. Fiber suspensions are definitely inhomogeneous and, as we shall see, behave according to a mechanism depending upon this inhomogeneity.

The Newtonian behavior is described by its linear

† The terminology commonly used in describing the various types of behavior may be misleading, since it often stems from a particular phenomenon exhibited by some fluid of a given rheological category, and has then come to be extended to all the other fluids of the same category. Thus not all the fluids in the dilatant classification exhibit the property of swelling when sheared.

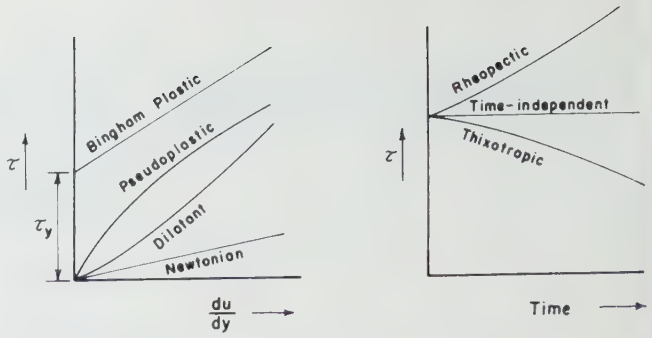


*Fig. 1. Types of rheological behavior*

proportionality between shear stresses $\tau$ and strain rates $du/dy$, or

$$\tau = \mu \frac{du}{dy} \tag{1}$$

where $du$ is the differential velocity increment over a distance $dy$ normal to the surface on which the shear stress acts, and the coefficient of proportionality $\mu$ is called the viscosity of the fluid, and is constant for a given fluid at a given temperature.

In order of increasing complexity from the Newtonian model, we list first the Bingham plastic, characterized by the equation:

$$\tau = \tau_y + \mu_s \frac{du}{dy} \tag{2}$$

where $\tau_y$ is the yield stress and $\mu_s$ is the slope viscosity, i.e., the slope of the $du/dy$ line (for $\tau > \tau_y$). Next come the pseudoplastic and the dilatant, characterized by the equation:

$$\tau = K \left(\frac{du}{dy}\right)^n \tag{3}$$

which is the equation of a straight line of slope $n$ and intercept $K$ in a log $\tau$ versus log $du/dy$ diagram; $n$ is dimensionless and $<1.0$ for the pseudoplastic, $>1.0$ for the dilatant; $K$ has dimensions which depend on the value of $n$. A single straight line of equation (3) actually fits only one portion of the logarithmic flow diagram of a pseudoplastic; outside of such portion, at lower and higher shear rates, the logarithmic diagram curves again.

At a boundary wall equation (3) becomes:

$$\tau_w = \left[K \left(\frac{du}{dy}\right)^n\right]_w \tag{3a}$$

In some cases $du/dy$ at the wall of a tube is proportional to the ratio of the maximum or mean velocity to the tube diameter. For Newtonian fluids in laminar flow, $du/dy$ at the wall $= 8V/D$. Using this we can express the wall shear as:

$$\tau_w = K' \left(\frac{8V}{D}\right)^{n'} \tag{4}$$

where $K'$ and $n'$ can be related to $K$ and $n$. The same relation resulted from analyses by Mooney and Rabinowitsch (*2, 3*).

Equations (1), (2), and (3) correspond to the following velocity distributions and shear stress relations:

*For Newtonian flow*

$$u = \frac{1}{4\mu} \frac{\Delta P}{L} (R^2 - r^2) \tag{5}$$

$$\tau_w = \mu \frac{8V}{D} \tag{6}$$

*For Bingham plastic flow*

$$u = \frac{1}{4\mu_s}\left[\frac{\Delta P}{L}(R^2 - r^2) - \tau_y (R - r)\right] \tag{7}$$

(valid for the portion of the cross-section where $\tau > \tau_y$, i.e., where $r > R_p = \tau_y 2L/\Delta P$, $R_p$ being the plug radius), and

$$\tau_w = \frac{\mu_s}{1 - 4/3(t_y/t_w) + (1/3)(\tau_y/\tau_w)^4} \frac{8V}{D} \tag{8}$$

*For Pseudoplastic or dilatant flow*

$$u = \frac{n}{n+1} \frac{1}{K^{n/1}} \left(\frac{\Delta P}{2L}\right)^{1/n} (R^{(1/n)+1} - r^{(1/n)+1}) \tag{9}$$

(which gives a profile blunter than the corresponding Newtonian if $n < 1$, and sharper if $n > 1$),

$$\tau_w = K\left(\frac{1+3n}{4n}\right)^n \left(\frac{8V}{D}\right)^n \tag{10}$$

The latter equation is not rigorous, because, as mentioned previously, equation (3) does not apply throughout the whole range of shear stresses in the cross-section.

A comparison of equation (10) with the summary equation (4) yields the following relationships between $K'$ and $K$:

$$K' = K\left[\frac{1+3n}{4n}\right]^n \left(\frac{8V}{D}\right)^{n-n'} \tag{11}$$

which, if $n' = n$, reduces to:

$$K' = K\left(\frac{1+3n}{4n}\right)^n \tag{11'}$$

A rearrangement of the Mooney and Rabinowitsch equation (*2, 3*) and a comparison with equation (3) leads (as shown by Metzner and Reed (*4*)) to the relationship between $n$ and $n'$:

$$n = \frac{n'}{1 - [1/(3n'+1)]\cdot(dn'/d\log\tau)} \tag{12}$$

which, if $n'$ does not vary with shear stress, reduces to $n = n'$. In principle equation (12) provides a means of evaluating $n$ in equation (3) from wall shear stress data such as given by pipe friction measurements. This conversion is meaningful only if the mechanism of shear transmission at a boundary is the same as through the interior of the fluid. For some suspensions segregation of the suspended phase causes differences, as will be discussed for fibers. In general, it is difficult to attribute a physical significance to $n$ and $K$ (or $n'$ and $K'$) when they are taken separately.

## 3. VISCOELASTIC BEHAVIOR

In addition to the rheological characteristics illustrated by the several types of flow curves, some fluids and suspensions exhibit elastic properties. Fiber suspensions develop fiber networks having elastic strengths, so that the suspension shows a viscoelastic behavior. To illustrate we will describe here some exploratory measurements on the rate and amount of settling, of the existence of elastic strain and on the motion decay time of a kraft pulp. A general review of viscoelastic behavior appears in (*5*).

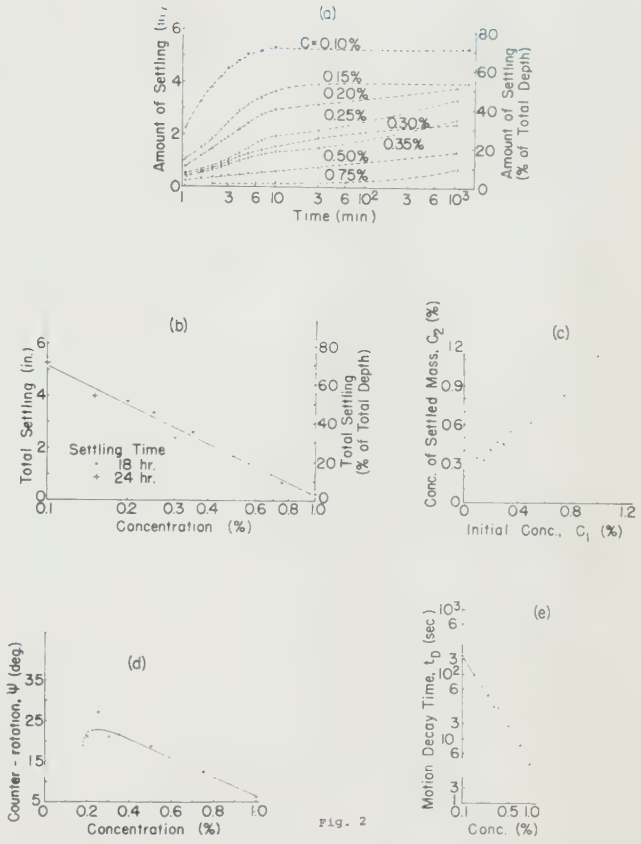


***Fig. 2. Settling and counter-rotation measurements—kraft pulp in cylindrical jars; jar diam. = 5.875 in.; liquid depth = 7.4 in.***

(a) Total settling vs. concentration. (b) Amount of settling vs. time. (c) Average concentration $C_2$ of settled mass vs. initial concentration $C_1$. (d) Elastic counter-rotation $\psi$ vs. concentration. (e) Motion decay time $t_D$ vs. concentration.

### *Settling*

The observation of fiber settling gives some insight into the structure of the suspensions. The data were taken with kraft pulp suspensions in cylindrical containers 5.875 in. diam. and filled to a depth of 7.4 in.

The suspensions were first stirred into a condition as uniform as possible, taking care to avoid rotatory motions; then, the rate and amount of settling were recorded (observing the position of the interface between the mass of fibers and the clear water above, and timing its descent). The results, plotted in Fig. 2 indicate that, within the range of concentrations tested, the networks formed by fiber interlocking (the presence of which is inferred from the counter-rotation tests described next) are not strong enough to support their own weight in the loose spatial configuration existing under fully dispersed conditions. This causes the fibers to settle into stronger, more compact masses. The amount of settling is obviously an inverse function of the compressive strength of the networks at different initial concentrations; it is shown in Fig. 2a to be a direct function of the initial concentration. The speed with which settling takes place is also an indication of the relative weakness (to compression) of the networks. The data shown in Figs. 2a and 2b cover observations of the amounts of settling over an arbitrarily chosen 18-hr. period (except—24 hr.—for the 0.10 and 0.15% concentrations); it is quite possible, however, that settling extends beyond such period, albeit at a very slow rate.

Fig. 2c shows the average concentrations $C_2$ of the settled mass (i.e., of the mass below the clear water layer) at the end of the observed period (18 or 24 hr.), as a function of the initial concentration. The concentration $C_2$ increases with $C_1$, indicating a progressive compression or change in texture of the settled mass as its height increases. (If $C_2$ were constant, independent of the initial concentration $C_1$, this would indicate that the settling of the fibers results in a mass of constant consistency and structure.)

***Counter-rotation***

Elastic counter-rotation is a phenomenon due to the setting up of elastic stresses in fiber suspensions, when sheared. The process whereby the stresses come into being can be interpreted in the light of the characteristic structure of the suspension as follows. Above a certain critical concentration, the individual fibers aggregate in clusters which, in turn, interlock in larger structures (fiber networks). These structures are not rigid, but possess a rather large amount of elasticity (or "springiness"), so that when the suspensions are subject to an external shearing action, a portion of the shearing force is absorbed by the structure itself, straining the bonds between fiber clusters and straightening individual fibers. This is analogous to the process of "retarded elastic response" occurring in the long-chain molecules of polymers (*5*). When the external shearing action ceases, the motion of the suspensions comes to a stop and then reverses its direction for a certain amount, until the internal elastic stresses are relaxed (i.e., until the bonds between fiber clusters and between fibers have reached again a configuration of least tension).

The counter-rotation of the suspensions examined was measured by setting them in rotatory motion in the same cylindrical container used for the settling measurements (and filled to the same height), letting the motion slow down to a stop and measuring the angle through which the suspensions would then rotate backwards, in direction opposite to the original one, before a permanent condition of rest would be reached. The amount of counter-rotation was found to be practically zero at concentrations below 0.20%, to climb to a maximum between 0.20 and 0.35%, and then to decrease in approximately linear fashion as concentration increased (Fig. 2d). From the results of the limited number of tests it seems reasonable to infer that the particular pulp tested has no interlocked structure at concentrations lower than 0.20%. The interlocked structure must form at approximately such concentration and be at first quite elastic, as the maximum in the amount of counter-rotation indicates. As concentration increases, however, the structure becomes denser and elastic deformation is progressively reduced. It is, of course, possible that counter-rotation takes place even when fibers do not interlock, i.e., at very low concentrations; but the effect must indeed be small as it can be caused only by the motion communicated to the suspending fluid through the bending or curling of the individual fibers rather than by extensive network deformations.

***Motion-Decay Time***

A second rheological parameter measured simultaneously to the angle of counter-rotation was the motion-decay time, or the time required for the subsidence of the rotatory motion imparted to the suspensions. As Fig. 2e shows, such time decreased exponentially with concentration, indicating a progressive increase in the viscous character of the suspensions.

## 4. LAMINAR FLOW OF FIBER SUSPENSIONS

The rheological models discussed in Section 2 describe laminar flow type conditions. Consequently, most attempts to use these models for correlation of experimental data and theoretical prediction of behavior of suspensions have been for the low velocity laminar or plug flow. In the following we will test these models against experimental laminar flow data and discuss differences between the actual flow mechanics and the model assumptions.

Figures 3 and 4 show flow resistance results for a fiber suspension in tubes. These data are for one of the pulps described in (*1*) and have general characteristics typical of all the fibers tested. This pulp is a kraft softwood designated as Long Lac 17. In Fig. 3 the measurements are plotted as wall shear stress $\tau_w$ versus the parameter $8V/D$ for both 2- and $^3/_4$-in. conduits. Data for the full range from the lowest laminar flow through the transition and turbulent flow regimes are shown. In general, the laminar flow regime is represented on the diagram by the area to the left of the dashed lines labelled "stability line." The transition and turbulent flow regimes fall in the area to the right of this line. The line shown represents a theoretical limit (to be discussed later) for laminar flow. As is seen, the beginning of the actual transition is approximated by this line. Data for the 2-in. conduit is shown in Fig. 4 plotted as pipe friction factor $f$ versus Reynolds number. In computing the Reynolds number the kinematic viscosity for the water phase is used. In this diagram the laminar flow regime corresponds to the lowest Reynolds number range covered for each concentration where the data are represented by a series of steep lines. In the definition sketches of Fig. 5 there is indicated for both types of diagram how the approximate extent of each regime can be indicated by a series of best-fit straight lines.

The laminar portions of the $\tau_w$ versus $8V/D$ diagrams can be considered as particular types of flow diagrams, relating shearing stresses at the wall to average rates of flow. If the ratio $V/D$ were directly proportional to the wall velocity gradient and if the mechanism of wall shear development were the same as in the fluid interior these diagrams would be interchangeable with the $\tau$ versus $du/dy$ representation. From the $\tau_w$ versus $8V/D$ diagrams the parameters $n'$ and $K'$ of equation (4) can be evaluated. The departure of $n'$ and $K'$ from 1.0 and $\mu$, respectively, indicated the degree of non-Newtonian behavior. Moreover, the extent to which $n'$ and $K'$ depend on the test conditions provides a means of testing the suitability of the various rheological models.

***Comparison with Rheological Models***

In systems for which the ratio $V/D$ is directly proportional to the velocity gradient at the wall, $K'$ and $n'$ should be independent of conduit size. On the other hand, consider systems in which the fluid displays a variable yield stress. Examples are a homogeneous material possessing a variable yield stress or an inhomo-

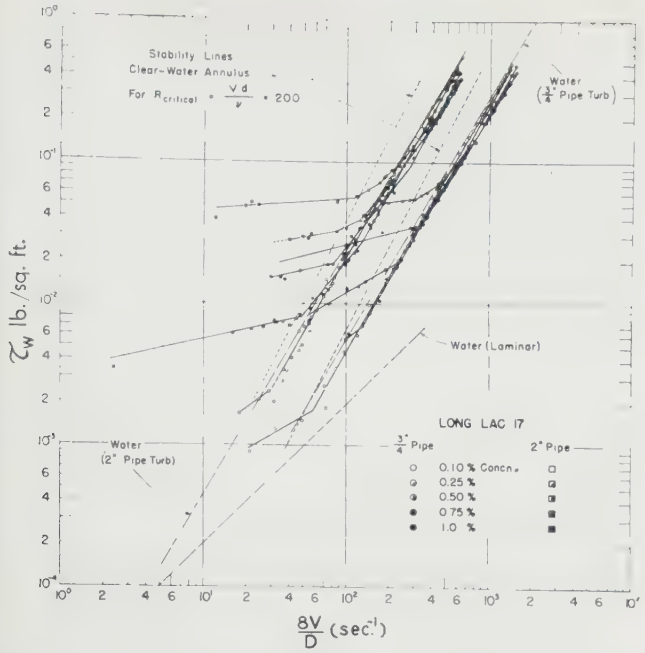


*Fig. 3. Log-log flow diagram: kraft softwood, Long Lac 17*

geneous suspension in which, by some mechanism of separation of phases, regions of different shear strength are developed. For these cases $V/D$ is not expected to be proportional to the velocity gradient at the boundary, and $K'$ and $n'$ in equation (4) can be expected to depend on pipe size.

Considering first the Bingham plastic [equation (2)], it can be shown that at the wall the shear stress is:

$$\tau_w = \mu_s \frac{8V}{D} \frac{1}{1 - (4/3)(\tau_y/\tau_w) + 1/3(\tau_y/\tau_w)^4} \qquad (13)$$

Since $n'$ in equation (4) is the slope of the log-log plot of $\tau_w$ versus $8V/D$, the particular value of $n'$ for which equation (4) also describes the wall shear stress for a Bingham plastic is obtained as:

$$n' = \frac{1 - (4/3)(\tau_y/\tau_w) + (1/3)(\tau_y/\tau_w)^4}{1 - (\tau_y/\tau_w)^4} \qquad (14)$$

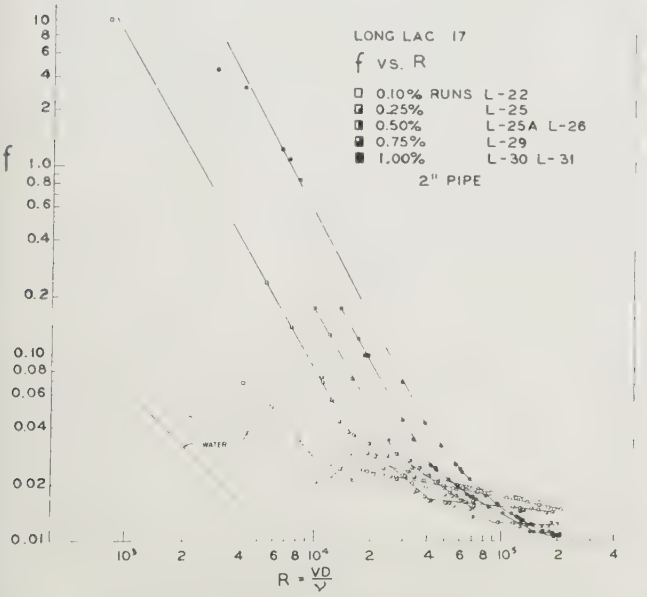


*Fig. 4. Friction factor vs. Reynolds number: kraft softwood, Long Lac 17*

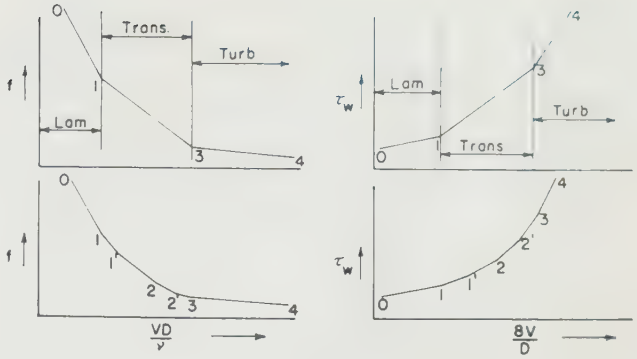


*Fig. 5. Definition diagrams—regimes of flow (log scales)*

Thus $n'$ now must be a constant for a particular ratio, $\tau_y/\tau_w$, otherwise the liquid cannot be termed a Bingham plastic. Furthermore, for a Bingham plastic $\tau_y$ is a constant and, for a given $\tau_w$, $n'$ should have the same value independent of conduit size. Figure 3 shows that the laminar test data, like those previously published (e.g., (*6*, *7*)) can be represented approximately by straight lines for each concentration and pipe size combination. Thus $n'$, measured as the slope of such a straight line, is constant over the entire range of laminar flow wall shear stress values. The conditions of equation (7) necessary for a Bingham plastic are not met, and we conclude that this model does not apply to fiber suspensions.

Baines's experiments (*6*) also showed that the size and velocity of the plug, and the slope of $f$ versus $VD/\nu$ did not agree with the Bingham plastic theory.

Values of $n'$ and $K'$ determined from $\tau_w$ versus $8V/D$ diagrams are shown in Fig. 6 for the three different wood pulps used in the experiments. It will be noted that for a given fiber and pipe size, $n'$ and $K'$ vary with concentration. $K'$ increases steadily while $n'$, after an initial drop, varies relatively little. The variation of $n'$ and $K'$ with concentration has been confirmed by Guthrie (*8*) from experiments extending up to 2%, and by Robertson and Mason (*9*) from experiments up to 0.98%. The latter report that for the higher concentrations $n'$ was approximately 0.25, a value roughly in agreement with the present experiments. It will be noted also that both $n'$ and $K'$ depend on the conduit

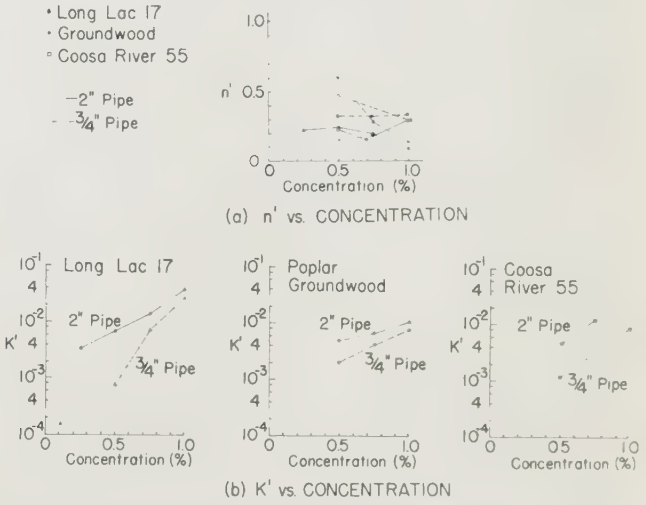


*Fig. 6. Rheological parameters n' and K' for laminar flow computed from friction loss data*

size. This is another indication that the Bingham plastic model is not satisfied.

The fact that the log-log flow diagrams are straight lines for the laminar regime suggests a pseudoplastic hypothesis (equation (3)). This is a more complex rheological model than the Bingham plastic, but it would still be a convenient one; first, because it lends itself rather easily to mathematical manipulations, and second, because it permits the derivation of generalized Reynolds number correlations from simple viscometer or pipe friction measurements.

Pseudoplastics have blunt velocity profiles (the blunter the profiles, the lower the value of the exponent $n$), which near the centerline are practically flat, very nearly resembling the plug of a Bingham plastic (as shown by the theoretical profiles in Fig. 7, computed using equation (9), from the actual values of $n'$ for two experimental runs). Unfortunately, the pseudoplastic model also has serious shortcomings. Like the Bingham plastic model, it cannot justify the variation of $n'$ with pipe size. It also gives a diameter of the flat velocity region which is much smaller than actually observed. The failure in predicting the variation of $n'$ with pipe size is indeed a major one from the viewpoint of pipe line design, because it makes it impossible to extrapolate to other conduit sizes the values obtained from measurements on a conduit or viscometer of given size.

The data in Fig. 3 also show that at the end of the laminar regime the critical value of the wall shear stress $\tau_w$ is roughly the same in the $^3/_4$- and 2-in. tubes. In general, critical wall shear depends on the conduit dimensions. This is true of Newtonian fluids where:

$$\tau_{w_{\text{crit.}}} = \frac{\text{const.}}{D^2} \qquad (15)$$

For a non-Newtonian fluid represented by equation (4), the transition to turbulence is reported (*4*) to occur at approximately constant values of the generalized Reynolds number $\mathbf{R}_L'$ (equation (25) to be discussed later). This results in:

$$\tau_{w_{\text{crit.}}} = \left(\frac{8\mathbf{R}_L'}{\rho}\right)^{n'/(2-n')} \frac{(K')^{2/(2-n')}}{D^{2n'/(2-n')}} \qquad (16)$$

so that the critical shear can be independent of $D$ only when $n'$ (and $K'$) obey a unique law of variation with $D$. Thus the condition of equal critical shear stresses is another factor inconsistent with the common rheological models.

There still remains the question of the possibility of a time dependency effect being present which is not covered by the time-independent models. A time-dependent stress would be reflected in a wall shear stress changing with distance in the flow direction. For stress relaxation the effect would be as shown by the dashed lines in Fig. 8a. Stress stiffening would give the dotted lines. Presumably, turbulence would set in (or disappear) when the proper combination of stress and effective dynamical viscosity was reached. The effective ratio of inertia forces to forces resisting nonturbulent shear would then be at the critical value. Consider flow rates just under the critical in the two pipes. In general, we would expect the steady-flow rate of head loss (or pressure drop) along a tube to depend on diameter. Then, for example, with the same average velocity $V$ in small pipe ($A$) and large pipe ($B$), the time-dependent effects would have to be opposite or of different magnitudes as in Figs. 8a, b, c to account for a convergence of the stresses.

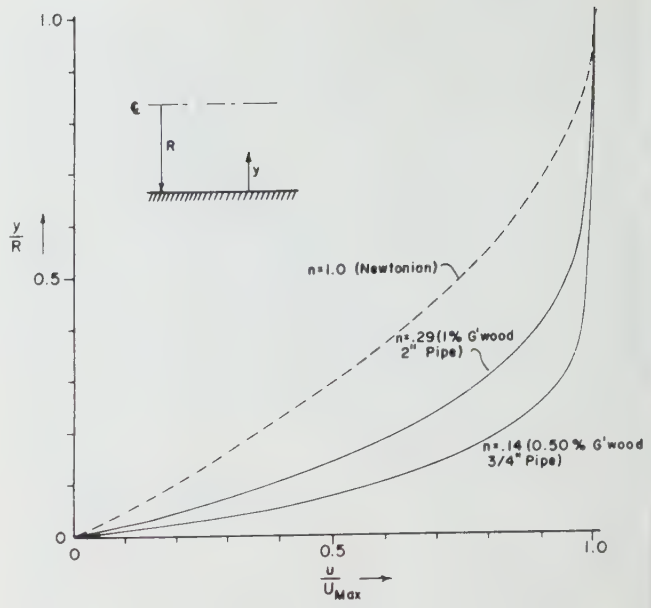


*Fig. 7. Pseudoplastic hypothesis: velocity profiles*

Effects such as shown in Figs. 8a, b, c were not observed in the pipe friction experiments. In all cases the head loss was proportional to distance along the pipe. Although the counter-rotation measurements previously described did indeed indicate the existence of elastic phenomena, there was no measureable evidence of the time-dependent effects of stress relaxation or stiffening.

### *Peripheral Annulus Model*

The common rheological models fail because they do not account for the fact that suspensions are not homogeneous but are two-phase systems in which segregation of the suspended phase can take place. This does take place in a special way under laminar flow conditions.

Visual observations show the existence of a peripheral water layer as observed by other investigators (*7, 10, 11*). The layer, in which most of the straining occurs, contains little or no fibers and surrounds a core where the fibers are concentrated. We have observed, in agreement with the findings by some of the cited authors, that the annulus formation is a dynamic phenomenon.

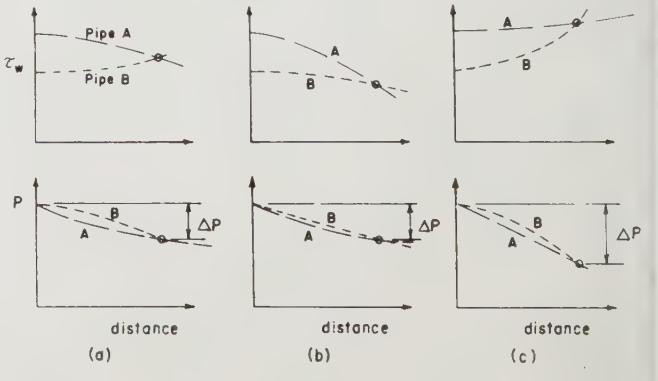


*Fig. 8. Time dependency effects in pipes for $\tau_{w\ \text{crit}}$ = const.*

(a) Stress relaxation vs. stress stiffening. (b) Stress relaxation at different rates. (c) Stress stiffening at different rates.

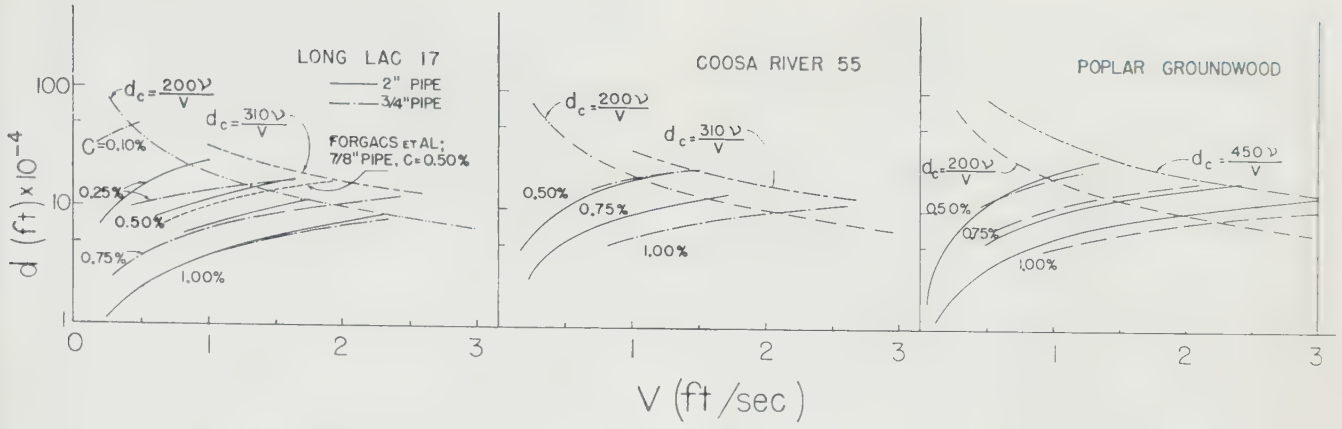


*Fig. 9. Computed water annulus thickness*

The annulus dimensions vary unsteadily and the mean thickness grows with flow rate. Fiber segregation and the presence of a clear water annulus is consistent with the fact that $n'$ does not vary with $\tau_w$ (i.e., $n'$ is constant over the entire laminar flow range), and does vary with pipe size. Moreover, it is consistent with experimental observations of the flow rate at which the laminar regime ends as a function of concentration and pipe size, and with the measured friction loss data.

If we now assume that, as visually observed, the annulus contains no fibers, that the whole velocity variation is confined to the annulus and, furthermore, it is linear (on account of the relatively small annulus dimensions), we can define, with Robertson and Mason (*9*, *12*), a Reynolds number for the annulus as:

$$\mathbf{R}_d = Vd/\nu \qquad (17)$$

where $V$ is the mean flow velocity (assumed equal to the velocity $U_p$ at the edge of the plug), $d$ the annulus thickness and $\nu$ the kinematic water viscosity. The overall resistance characteristics of the flow in the pipe should then be governed by this annulus Reynolds number. In Fig. 3 lines have been drawn corresponding to a critical value of $\mathbf{R}_d = 200$. It is seen that, in general, the end of the laminar regime occurs in the neighborhood of the points where the $\tau_w$ lines cross the constant $\mathbf{R}_d$ lines. This is a further confirmation of the role played by the conditions in the annulus, and in general, of the soundness of the clear annulus hypothesis. A detailed analysis of the transition process will be given later. Reverting to the flow conditions before transition, the previous assumptions that the velocity in the annulus is linear, and $V = U_p$, lead to a shear stress at the wall given by:

$$\tau_w = \mu\, V/d \qquad (18)$$

whence, as shown by Richardson (*11*) and Forgacs, *et al.* (*7*):

$$d = \mu \frac{V}{\tau_w} = \frac{V\mu}{f\, V^2/8} = 8\, \nu/Vf \qquad (19)$$

The $d$ values resulting from equation (19) are plotted in Fig. 9 for the three wood pulps tested in the MIT experiments (*1*).

It is seen that $d$ increases with velocity, decreases with concentration, and for the same concentration and velocity, is nearly equal in the two pipes. It is also seen, that, other things being equal, $d$ is smaller for the longer fibers. This is consistent with the view that such fibers form stronger plugs, extending further from the centerline toward the wall. As a result of the smaller annulus thicknesses, the velocity variation is confined to a narrower region, and for the same average velocity, a higher shear stress at the wall ensues.

For comparison purposes, the $d$ values computed by Forgacs, *et al.* (*7*) from experiments with bleached sulfite fibers at 0.50% concentration in a $^7/_8$-in. pipe have been plotted in the diagram for Long Lac 17. The agreement with the present results is good.

In the $d$ versus $V$ representation, a hyperbola of equation $d = \text{Const.}\ \nu/V$ would correspond to a constant $\mathbf{R}_d$ value. It is seen that both for Long Lac 17 and Coosa River 55 the region where the laminar regime ends can be delimited by two lines corresponding to $\mathbf{R}_d = 200$ and $\mathbf{R}_d = 310$. For poplar groundwood an average upper value of approximately 450 is more satisfactory (except for the 1.0% concentration in the 2-in. pipe, which has a transition $\mathbf{R}_d$ of approximately 530). These computed values depend, of course, on the viscosity assumed. It is possible that the high values for groundwood are due to "fines" causing the annulus viscosity to exceed the clear water viscosity.

Richardson (*11*) and Robertson, *et al.* (*9*), have described a mechanism suggesting deflection of fiber ends protruding from the central plug as the explanation of the annulus formation and the increasing annulus thickness with velocity. Robertson, *et al.* (*9*), also suggest the possibility of elastic elongation of the plug under shear stress. In general, we agree with these notions but propose an amplified model of the flow process to

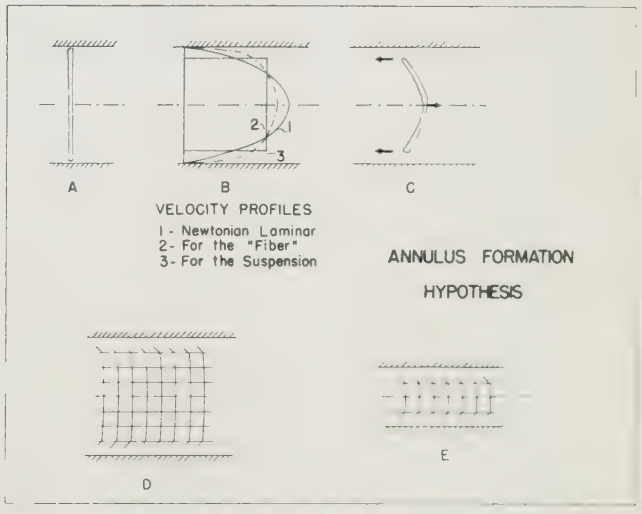


*Fig. 10. Annulus formation hypothesis*

include description of the effect of fibers on velocity profile as well as on the annulus formation.

Let us consider an ideal fiber oriented with its longitudinal axis normal to the axis of the pipe, as shown in Fig. 10A. In the absence of the fiber, the velocity distribution of the Newtonian suspending phase is the usual parabolic one for laminar flow. The presence of the fiber alters this original velocity distribution which, in turn, interacts with the fiber and deforms it. Drag forces arising from velocity differences between the liquid medium and the fiber (which can move only at one velocity once it has reached equilibrium configuration) will arch the fiber as shown in Fig. 10C. Thus, a gap is formed between the fiber tips and the wall, which can be expected to be a direct function of the fiber flexibility and of the rate of flow (i.e., of the magnitude of the drag forces on the fiber). Concurrently with deforming the fiber, the drag forces modify the original velocity profile, which now becomes flatter in the central portion of the cross-section, and steeper near the boundary (Fig. 10B). This results in a higher wall shear, and hence higher friction losses.

We can now imagine a network as being formed by a very large number of fibers, all cross-connected, as shown in Fig. 10D. Depending on the concentration, it will possess different degrees of elasticity (as inferred from the counter-rotation measurements). At low concentrations the network deformation, and its effect on the velocity profile, should be similar to the case of the idealized single fiber. As concentration increases, the voids between the fibers decrease, so that the velocity profile should become increasingly blunt. Therefore, for a given flow rate, the wall shear stress increases, as indeed the friction loss data indicate. Simultaneously, at higher concentrations the network becomes less elastic (see Fig. 2D), and the fiber deflection should be confined mainly to the loose fiber ends at the peripheral annulus. For a given suspension and plug velocity (which is nearly equal to the average velocity), the annulus should then have the same dimensions in pipes of different sizes, as suggested by Figs. 10D and 10E. The small differences in the computed annulus thickness plotted in Fig. 9 appear indeed to verify this view.

## 5. TRANSITION REGIME

The transition regime has been defined as the regime intermediate between the conditions characteristic of low flow rates, when the flow is laminar in the annulus, and those characteristic of large flow rates, when the flow appears to be fully turbulent throughout the cross-section.

The main questions concerning the transition are:

1. When does the flow cease to be laminar in the annulus and the transition regime begin?
2. What are the flow characteristics in the transition regime?
3. Over what range of flow rates does the transition exist before turbulence eliminates the flocculated plug core completely?

### *Onset of Turbulent Conditions in the Annulus*

It has been pointed out that the experimental data are consistent with the view that the laminar flow in the annulus becomes unstable when a critical value of the annulus Reynolds number is reached. Let us discuss the probable magnitude of such critical Reynolds number. Robertson and Mason (*12*) have pointed out that the flow could be approximated by a Couette flow between parallel surfaces for which Orr and Hamel have obtained theoretical values of 177 and 288, respectively (*13*). The value of 200 used in the $\tau_w$ versus $8V/D$ diagrams has been selected because it falls between the two above values, and also falls close to the experimentally determined critical Reynolds number for the transition in the gap between an outer stationary cylinder and an inner concentrically rotating cylinder (*14*). Forgacs, *et al.* (*7*), have reported an average value of approximately 310, from flow experiments with dilute fiber suspensions. This value is shown in Fig. 9 to represent satisfactorily the upper limit of the $\mathsf{R}_d$ values at which transition takes place in the present experiments with Long Lac 17 and Coosa River 55. With poplar groundwood a higher upper value of 450 is more satisfactory.

The Couette flow hypothesis in the case of the peripheral annulus is, of course, only a rough approximation of the true physical picture. In the first place, the upper boundary of the flow in the annulus, i.e., the edge of the plug, is not well defined, and is very irregular, so that the annulus thickness, $d$, has only a statistical meaning. In the second place, such a boundary is elastic and porous, in the sense that it can deform freely and allows exchanges of fluid masses between the annulus and the plug. Then the critical Reynolds number must be expected to vary with concentration, type of fiber and also, probably, fiber length to pipe diameter ratio, i.e., with all those factors which may affect the characteristics of the upper boundary. Also, once turbulence sets in, its intensity and scale—and hence the velocity profiles and energy dissipation in the annulus—can be expected to be affected by the characteristics of the plug, before the turbulence succeeds in destroying the plug itself.

In the $\tau_w$ versus $8V/D$ correlation, lines of constant $\mathsf{R}_d$ are parallel to the dashed "stability" lines shown in Fig. 3 for $\mathsf{R}_d = 200$. In the $f$ versus $\mathsf{R}$ correlation of Fig. 4, a constant $\mathsf{R}_d$ value corresponds to a constant $f = 8/\mathsf{R}_d$. Thus, we can see, from either correlation, as well as from Fig. 9, that in our experiments $\mathsf{R}_d$ did not have a unique value at all the transition points, but indeed varied with fiber, concentration, and pipe size.

It must be noted that the concept of a transition governed by a critical $\mathsf{R}_d$ needs not to be in conflict with the observation of a constant shear stress at transition in the two different pipes, for a given suspension. It merely imposes a further specification on the conditions at transition. The strictest specification would occur if the critical $\mathsf{R}_d$ values and also the critical $\tau_w$ values were exactly equal in the two pipes. Then the products $Vd$ should be equal, and so should the ratios $V/d$. Both conditions could be satisfied only if the $V$ values and the $d$ values were the same, i.e., if there were perfect identity in the flow conditions in the annuli of both pipes.

A further insight into the onset of turbulence in the annulus is perhaps offered by the following considerations. The laminar sublayer thickness $\delta'$ for Newtonian turbulent flow in smooth pipes may be expressed as:

$$\delta' = \frac{C\,\nu}{(\tau_w/\rho)^{1/2}} \qquad (20)$$

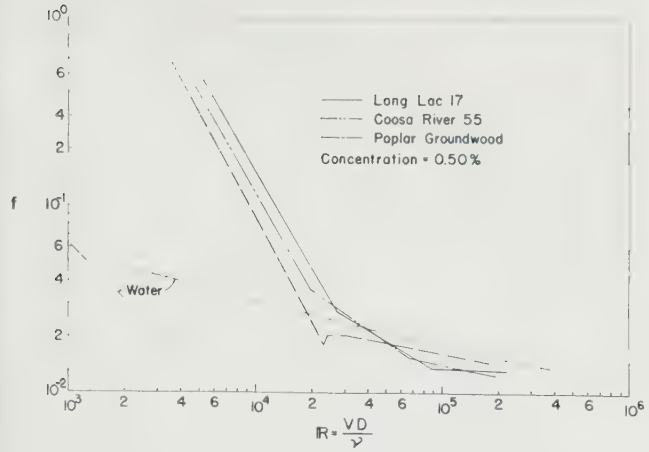


*Fig. 11. Comparison of friction loss data showing dependence of transition on fiber type*

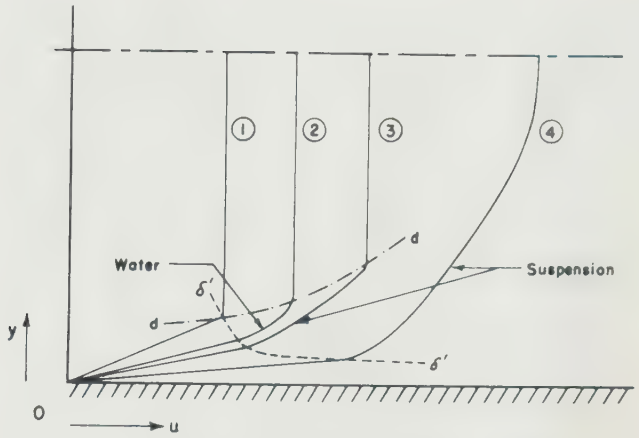


*Fig. 12. Hypothetical velocity profiles: transition from laminar to turbulent regime*

with the constant $C = 11.6$. The concept of $\delta'$ is a statistical average. Equation (20) gives the distance from the boundary within which turbulent conditions do not set in. We could then be tempted to extend this meaning of $\delta'$ to conditions other than those for Newtonian turbulence in pipes if, however, the velocity distributions near the boundary were approximately similar, i.e., linear in both cases. Of course, the values of the constant in equation (20) should be expected to be different for different turbulence conditions outside the sublayer, since equation (20) assumes that the turbulent field is specified only by $\tau_w$.

It is now interesting to note that if we were to plot logarithmically the computed annulus thicknesses, $d$, at transition versus the corresponding wall shear stresses $\tau_w$, we would find them to fall in a band delimited by two parallel lines of equation (20), with the constants equal to 13 and 24, respectively. This would then indicate that the above described concept of $\delta'$ as a stability boundary also applies to the flow in the annulus, which becomes turbulent when $d > \delta'$. The influence of the characteristics of the region outside the annulus (variable with type of fiber, concentration and also pipe size) would be accounted for by giving different values to the constant. This would also mean that when turbulence sets in the annulus, it should be limited to the portion of the annulus where $d > \delta'$, i.e., to the region near the plug.

If we combine the critical annulus thickness given by an equation of the type of equation (20) with that resulting from $\mathsf{R}_d$ critical $= Vd/\nu$, we find that the constant $C$ of equation (20) is related to $\mathsf{R}_d$ critical and to the friction factor at transition by the following expression:

$$C_c = \frac{\mathsf{R}_{d_c}\sqrt{f_c}}{\sqrt{8}} \qquad (21)$$

or, since $\mathsf{R}_{d_c} = 8/f_c$

$$C_c = \sqrt{\frac{8}{f_c}} \qquad (22)$$

In conclusion, the end of the laminar regime in the pipes is explained with the onset of critical conditions in the annulus. The onset can be described in terms of a critical value $\mathsf{R}_{d_c}$ of the annulus Reynolds number, or of a critical annulus thickness $d_c$ given by an equation of the type of equation (20). The numerical values of these critical parameters, however, fluctuate with type of suspension and pipe size. Only as a rough approximation can the transition be predicted on the basis of a constant value of $\mathsf{R}_{d_c}$ or $d_c$ (i.e., of $f_c$, from the preceding equations), for all cases. In the present experiments $f_c$ varied approximately between 0.045 and 0.015, corresponding to $\mathsf{R}_{d_c}$ values of 178 and 532. These are, however, extreme values for the whole range of conditions tested, and may be affected to a certain extent by the difficulties in determining exactly the transition points. Thus, as shown by Fig. 9, we can say that on the average the transition occurred in the $\mathsf{R}_d$ range between 200 and 310 for the longer fibers (Long Lac and Coosa River), and between 200 and 450 for the shorter fiber (groundwood).

### *Flow Characteristics of the Transition Regime*

The type of fibers influences not only the onset of the transition to turbulence but the character of the transition regime. This is shown clearly by the comparisons in Fig. 11. For the longer wood pulp fibers (Long Lac 17 and Coosa River) the transition extended over a wide range of flow rates (regime 1–3 in Fig. 5). For the short groundwood fibers the transition occurred rather abruptly.

Although we have no measurements indicating the velocity or other characteristics of the flow in the annulus during the transition, it seems apparent that it is turbulent. This has been recognized by Robertson and Mason (*12*) and Forgacs, *et al.* (*7*), who suggest that the water layer becomes turbulent and, with increased velocity, turbulent stresses rise sufficiently to disrupt the plug. Let us examine the probable mechanism in more detail. In the preceding section the concept of a critical distance from the wall within which—statistically—the flow is not turbulent, was exposed. From such concept it would follow that turbulence is likely to set in where the annulus thickness exceeds the critical distance $\delta'$, i.e., near the plug rather than near the solid boundary. With the help of Fig. 12 let us consider the steps of the resulting transition process.

At the very beginning, the turbulent intensities are necessarily low (on account of the narrow space to which the process is confined), so that momentum transfer is weak. The velocity profile in the space between the laminar sublayer and the plug, if it were represented by a power law, $u/U_p = (y/R)^{1/m}$, would

have a low value of $m$. Turbulence pushes the outer edge of the laminar sublayer closer to the wall. This results in a steeper velocity profile near the wall and thus a higher head loss than if the flow were laminar throughout the annulus (curve 2 in Fig. 12).

As the flow rate increases further, the annulus becomes larger, at the expense of both the laminar sublayer and the plug, which is more and more compressed. The consequent increase in dimensions of the turbulent portion, and the increased shear, result in an increasing amount of turbulent fluctuations (and the turbulent velocity profile could probably be represented by a higher value of $m$ in the power law).

The next stage in the transition process (curve 3 in Fig. 12), occurs when the shear stresses at the surface of the plug have reached a level high enough to cause the progressive disruption of the plug, from the surface inward. The space between the laminar sublayer and the new, ever receding surface of the plug, is now occupied by a mixture of water and fibers in turbulent motion. Since, as will be discussed in a later article, turbulence measurements have shown the presence of fibers to have a damping effect on the turbulent level, the turbulent agitations in this annular space should be lower than those corresponding to the previous phase, when the space was occupied by water only. The velocity profile should probably become less steep again (because the momentum transfer is lower) and could be represented by a lower value of $m$. The laminar sublayer, however, is smaller, so that the velocity profile at the wall is steeper (and hence, the head loss is higher) than for the conditions represented by curve 2 in Fig. 12, at the same flow rate.

Finally, with the increase in flow rates, a point is reached where the regime becomes turbulent throughout the cross section (curve 4 in Fig. 12). The higher shear stresses and the larger space now available for the development of turbulent vortices induce a higher level of turbulent agitation and thus a better momentum transfer. This gives steeper velocity profiles and still higher head losses.

The change in turbulent characteristics from the previous stage (represented by curve 3 in Fig. 12) could be expected to be rather sudden, since the disappearance of the plug doubles the transverse dimensions available to the turbulent vortices; this could be expected, in turn, to be reflected in a rather abrupt change in the head loss characteristics. On the other-hand, at the higher concentration even under fully developed turbulent conditions, the flow probably retains a pluglike character near the centerline (albeit only statistical in nature, the flocs associating and dissociating continuously). Thus, the situation would actually not be very different from the last phase of the transition, when a permanent plug was present. This would tend to lessen and make less abrupt the change in head loss characteristics and to make more difficult a determination of the exact rate of flow at which turbulence sets in over the whole cross-section.

In the light of the mechanism and sequence just proposed, the following explanation is offered of the experimental trends observed in the transition region:

(a) Groundwood, characterized by very short and relatively inflexible fibers, forms a rather weak plug, which is destroyed rapidly under the action of turbulence

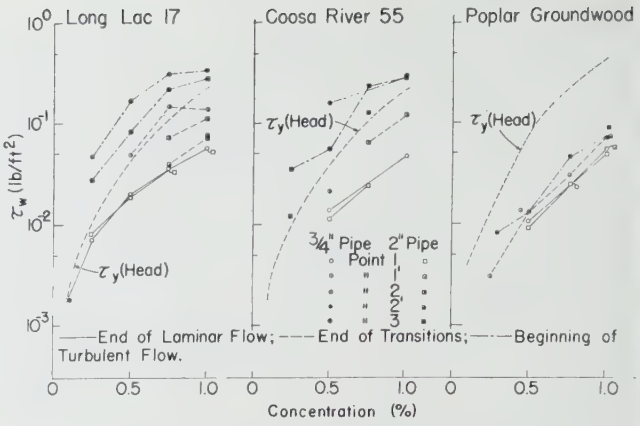


*Fig. 13. Transition wall shear stresses*

in the annulus and of high shear stresses. Thus, the transition is accomplished within a small range of flow rates, and sometimes is almost instantaneous. With Long Lac 17 and Coosa River 55, which have longer, more flexible fibers, and form stronger flocs, the transition process is more gradual, and at the higher concentrations all the stages outlined are probably well represented. The reduction of the number of intermediate transition stages at the lower concentrations is attributed to the weaker plug structure at such concentrations, so that the disruption process takes place very rapidly.

(b) The exact point at which the disruption of the plug begins cannot be isolated with certainty in the present experiments, because quantitative measurements of the plug yield stresses of our suspensions are not available. Let us assume the wall shear stress equals the shear stress at the plug surface, which is nearly so for small annulus conditions. We can determine the wall shear stress at transition from diagrams like Fig. 3. Figure 13 shows such values for the three wood pulp fibers. Using this figure and with reference to the definition sketch of Fig. 5, we could then hazard the guess that the yield stress is reached at transition point 2 or, in its absence (mainly at the lower concentrations), at transition points 1 or 1′. The fact that with Long Lac 17 the wall shear stress at point 2 appears to be higher in the $^3/_4$-in. pipe than in the 2-in. pipe needs not to be in conflict with the above hypo-

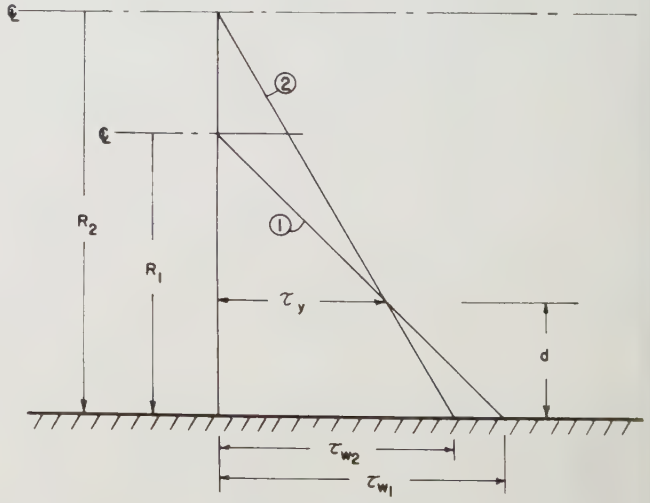


*Fig. 14. Shear stress diagrams at transition for same $\tau_y$ in $^3/_4$ and 2 in. tubes*

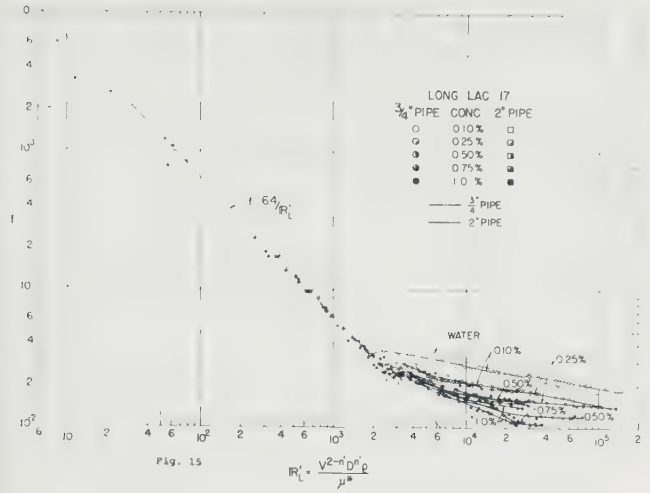


*Fig. 15. Friction factor vs. generalized Reynolds number—kraft softwood, Long Lac 17*

thesis, as long as the annulus thicknesses in the two pipes remain not very different in the transition region (as suggested by the shear stress sketch in Fig. 14). From Fig. 13, we must also infer that the yield shear stress for groundwood is lower than for the other two pulps, since in general, the transition for groundwood sets in and is accomplished at appreciably lower wall shear stresses. This is not in agreement with an empirical expression proposed by Head in 1952 (*15*), and represented by the dashed lines in Fig. 13, according to which suspensions of shorter fibers have higher yield stresses than suspensions of longer fibers. In a subsequent study, Head and Durst (*16*) reported yield stress data obtained with a more refined method, which show that at the lower concentrations the yield stress of groundwood suspensions becomes lower than for suspensions of other fibers. Unfortunately, their data refer to a much higher range of concentrations than in our experiments, and cannot be safely extrapolated. Richardson (*11*), using a wide-gap rotational viscometer, measured yield stresses of the plug under steady-flow conditions (i.e., with a method inherently more accurate than the original one used by Head, who measured $\tau_y$ under unsteady conditions). This resulted in lower yield shear stresses than those given by Head's original expression, but these data were also for high

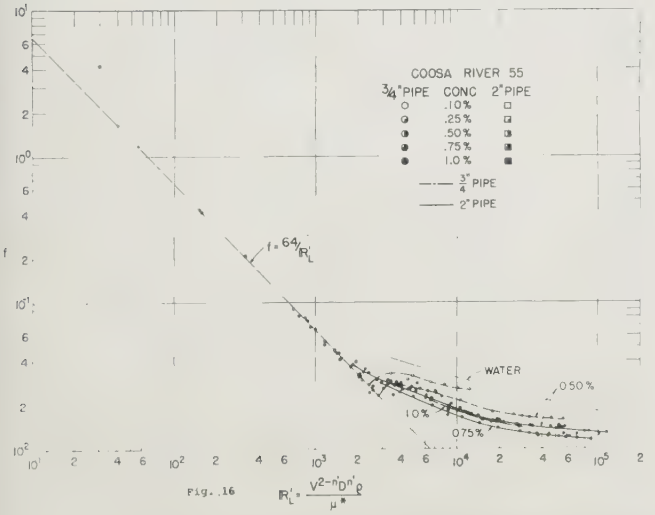


*Fig. 16. Friction factor vs. generalized Reynolds number—kraft softwood, Coosa River 15*

concentrations and cannot be extrapolated to our range.

(c) The significance of the region 1–1′ in Fig. 5 is not very clear. In some cases, best fit lines could have been drawn from the beginning of the laminar regime all the way to point 1′, but in some other cases the change in slope at point 1 is quite appreciable. It may also be that the region 1–1′ corresponds to turbulent conditions in the annulus, but in the absence of velocity distribution measurements in the annulus, this is a futile speculation.

The "valleys" in the friction loss diagrams at the end of the laminar regime have probably the same significance as the transition region between laminar

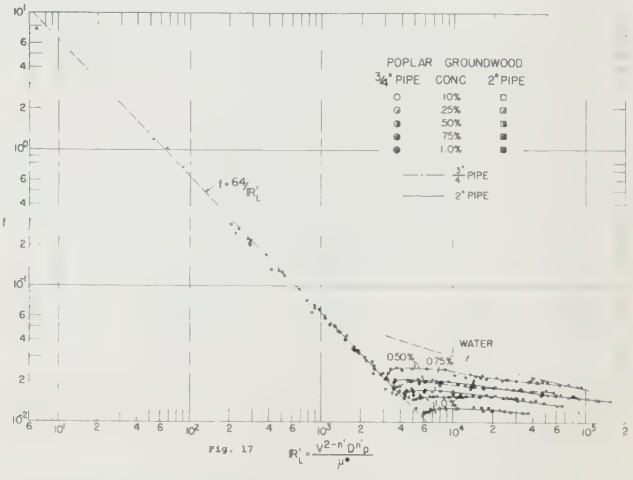


*Fig. 17. Friction factor vs. generalized Reynolds number—poplar groundwood*

and turbulent Newtonian flow. They are more noticeable for groundwood than for the other suspensions. In general, the data for groundwood show less scatter, which suggests that the flow is more stable.

(d) In summary, the gradual increase in the slope $n'$ of the wall shear versus velocity diagrams (or the flattening of the $f$ versus **R** slopes) follows from the proposed mechanism for the transition. Essentially, the transition marks the evolution from a system containing no turbulence to a system wholly turbulent. This occurs through stages in which turbulence spreads to progressively larger portions of the cross section, so that there is a progressive increase in the rate of energy dissipation.

### *End of the Transition Regime*

In the preceding it was proposed that the end of the transition occurs when the plug is completely disrupted, so that turbulence spreads over the whole cross-section. On account of the possible existence of pluglike fiber associations near the centerline even under turbulent conditions, the end of the transition regime cannot always be expected to be marked by a very sharp change in the slope of the friction loss diagrams. Here we will limit ourselves to observe again that for groundwood, on account of its weak plug structure, the end of the transition occurs very close to its inception. For the other fibers the end of the transition occurs at much higher flow rates; the process is gradual and, especially in the case of Coosa River, the end of the transition cannot be determined with certainty.

## 6. GENERALIZED CORRELATIONS

Recently generalized Reynolds numbers have been used to correlate the friction factors of non-Newtonian fluids. These generalized Reynolds numbers are obtained by forcing a fit between expressions for the friction factor, $f$, obtained experimentally, and expressions for $f$ valid for Newtonian fluids. Thus Metzner (4) equates the defining relation:

$$f = \frac{\Delta P}{\gamma(L/D)(V^2/2g)} = \frac{8\tau_w}{\rho V^2} \tag{23}$$

to the Poiseuille value for Newtonian laminar flow, $f = 64/\mathbf{R}$, and obtains:

$$\mathbf{R} = \frac{8\rho V^2}{\tau_w} \tag{24}$$

Then, using equation (4) a generalized Reynolds number $\mathbf{R}_L'$ is obtained as:

$$\mathbf{R} = \mathbf{R}_L' = \frac{V^{2-n'}D^{n'}\rho}{K'8^{n'-1}} \tag{25}$$

Correlation by this parameter should be good if $K'$ and $n'$ are constants, as they are for suspensions over the laminar flow range. Figures 15 to 17 are plots of the experimental friction factors from the MIT tests versus the generalized Reynolds number $\mathbf{R}_L'$. The experimental data correlate well in the laminar range, but depart from the curve of forced fit when the flow becomes turbulent. The turbulent water curve is not followed and the values for different concentrations and conduit sizes diverge.

On the other hand, it is pertinent to emphasize at this point that the Metzner type correlation can be obtained whenever the friction loss data can be represented by a power law whether the flow is laminar or turbulent, that is, whenever $K'$ and $n'$ are constants, or alternately, whenever the friction factor, $f$, can be expressed in terms of some power of Reynolds number. For example, if we represent the friction factor for Newtonian fluids by a power law:

$$f = \frac{C}{\mathbf{R}^{1/m}} \tag{26}$$

we can use this as the basis of reference instead of the Poiseuille laminar flow relation to obtain:

$$f = \frac{8}{\rho V^2} K' \left(\frac{8V}{D}\right)^{n'} = \frac{C}{\mathbf{R}^{1/m}}$$

or

$$\mathbf{R} = \left[\frac{C}{64} \frac{V^{2-n'}D^{n'}\rho}{K' 8^{n'-1}}\right]^m$$

$$\mathbf{R} = \left[\frac{C}{64}\mathbf{R}_L'\right]^m \tag{27}$$

Thus, in principle, we can use the single parameter $\mathbf{R}_L'$ for correlating all power law friction factor data. It follows that since the whole range of fiber suspension friction loss data can be represented by a series of particular combinations of constant values of $K'$ and $n'$, a continuous correlation can be achieved if the appropriate values of $K'$ and $n'$ are used for each individual regime. This serves very little practical purpose, however, since from an engineering viewpoint such correlations are useful to the extent that $K'$ or $n'$ are known, or easily obtained (and this is not the case for transition and turbulent conditions), so that pressure drops, limits of each flow regime, etc., may be predicted from a generalized Reynolds number plot.

In the case of fiber suspensions, the situation is further complicated by the dependence of $n'$ and $K'$, even under the equivalent of laminar conditions, not only on kind of fiber and concentration, but also on pipe size, as discussed already. Thus even if $n'$ and $K'$ were obtained from experiments in a given channel, the values cannot be applied, through a scaling-up procedure, to the flow in channels of different sizes.

## 7. SUMMARY OF FINDINGS AND CONCLUSIONS

The experimental findings bearing on the mechanism of laminar shear flow and its transition to turbulence include:

1. Wood pulp and nylon fibers show viscoelastic properties in simple tests for concentrations above a certain magnitude (0.2% for the kraft pulp examined in detail).

2. Typical flow resistance data for the laminar flow range of wood pulps show,

(a) The head loss is proportional to length of conduit for a fixed flow rate.
(b) The head loss over a given length exceeds that for water (Newtonian) at the same laminar flow rate.
(c) The head loss over a given length is proportional to the flow rate raised to a fractional power as compared to being directly proportional to flow rate for Newtonian fluids.
(d) The type of transition to turbulence depends on the type of fiber.
(e) The numerical values of the critical wall shear and velocity at the transition to turbulence are approximately independent of conduit size, but depend on type of fiber and concentration.
(f) If the relation between wall shear and flow rate is expressed by:

$$\tau_w = K' \left(\frac{8V}{D}\right)^{n'}$$

the data show that $K'$ and $n'$

(1) Vary with concentration, and
(2) Vary with conduit size, but
(3) Are independent of flow rate up to the transition to turbulence

Analysis of the experimental results leads to the following observations and conclusions:

1. Dilute fiber suspensions in laminar flow represent a particular type of non-Newtonian flow characterized by a macroscopic suspended phase forming extended elastic networks through mechanical entanglement.

2. Because the common rheological models do not account for nonhomogeneity they fail to describe the flow of fiber suspensions.

3. Owing to the two phases and the segregation of the suspended phase, the mechanism of shear transmission varies across the flow section. The means of shear transmission is not the same at the wall and in the central portion of the flow.

4. Viscoelastic properties play a role although there was no observed evidence of time variations such as stress-relaxation or stress-stiffening.

5. The well-known clear water annulus model does agree with present results, and previously published models are amplified to explain how elastic fiber networks interact with fluid shear and drag to modify Newtonian velocity profiles and produce phase separation leading to annulus formation.

6. The Couette flow hypothesis for flow in the clear water annulus is approximately correct in describing the mean conditions during laminar flow.

7. The Couette flow hypothesis provides an approximate physical explanation of the critical conditions for transition to turbulence. However, the transition cannot be described by a simple parameter as it depends on type of fiber and concentration.

8. Use of the laminar sublayer $\delta'$ in a criterion for transition in the annulus is suggested.

9. The sequence of events leading from the laminar to the turbulent regime is interpreted in terms of the velocity profile and annulus thickness.

10. Metzner type generalized correlations of flow resistance data are of limited usefulness because the parameters in the correlating Reynolds numbers are neither constant for a given fiber nor easily obtained.

Finally, it should be noted that the term dilute suspension to which this discussion has been directed applies to a limited range of concentrations. There is a lower limit below which fiber interlocking and network formation will not occur. Above some upper limit the networks become very dense and possibly less subject to elastic deformation.

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# The Chemical Composition of the Newly Formed Inner Bark of Douglas-Fir

G. W. HOLMES and E. F. KURTH

**An examination of the extractives from the newly formed inner bark of Douglas-fir revealed that the ether solubles contained predominantly *d*-catechin and *l*-epicatechin. After ion-exchange separation, the water solubles were examined paper chromatographically. The amino acids, glutamic acid, aspartic acid, serine, glycine, proline, alanine, threonine, valine, leucine, and isoleucine were shown to be present. Some evidence was also obtained for the presence of tyrosine and phenylalanine. Sucrose, fructose, and glucose were present in varying amounts during the year, but raffinose appeared to be present only during the growing season. In addition to six unidentified hydroaromatic substances, quinic and shikimic acids were present. An unidentified, Wiesner-positive material was found which may be a wood or bark lignin precursor.**

**The presence of the catechins in the living bark implies that they are the precursors of the polyphenol components of either the bark or wood. This agrees with the general theory that many of the high-molecular-weight polyphenols—tannins, phlobaphenes, and possibly bark lignins—are polymers of catechins and related substances. The absence of catechins in the nonliving portion of the outer bark further substantiates this theory. The presence of shikimic and quinic acids, intermediates between sugars and aromatic compounds, are also believed to be precursors of lignin and flavonoids. The amino acids present appear to be primarily responsible for protein synthesis, rather than the synthesis of structural components of the tree.**

IN RECENT years a considerable amount of interest has been centered on the chemical composition of conifer barks. Several studies have been made of the extractives of Douglas-fir bark, and the polymeric polyphenols of this bark have also been characterized.

The bark lignins of Douglas-fir have been shown by Kurth and Smith (*1*) to differ from wood lignin in several respects. The bark lignins generally are lower in methoxyl content than wood lignin, and in addition, carboxyl groups have been shown to be present in isolated bark lignins, whereas these groups have not been detected in wood lignin.

It was felt that additional knowledge concerning the structure of the bark lignins would be valuable. Since most of the previous work had been of a degradative nature, it was believed that a different approach might be desirable. Examination of the substances in or near the cambial area could reveal the presence of any precursors of the bark. It is obvious that any substance in this area may be a precursor of either the wood or the bark, or neither. However, it was believed that an examination of the extractives from the newly formed inner bark might present some new and valuable information concerning the biosynthetic pathways of the tree.

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Table I. Yields of Extractives from the Newly Formed Inner Bark

Percentages based on moisture-free, unextracted bark

| *Date sample collected* | *Hexane solubles* | *Methanol solubles* | *Water solubles* | *Ethyl ether solubles* |
|---|---|---|---|---|
| June 6, 1959 | 1.21 | 0.75 | 31.2 | 0.92 |
| July 21, 1960 | 1.57 | 0.93 | 42.0 | 0.74 |
| Sept. 23, 1959 | 2.20 | 1.13 | 39.8 | 0.69 |

It is also possible that any information concerning the chemistry of the cambial tissue may throw some light on the preferential-attack phenomenon of Douglas-fir bark beetles. These beetles appear to attack and kill first the damaged or unhealthy trees. It would seem that the chemical composition of the cambial area might be the key to the resistance or susceptibility of any certain trees to the beetles.

## COLLECTION AND PREPARATION OF MATERIALS

The samples used in this work were collected near Corvallis, Ore., in the Woods Creek area. Most of the work reported herein was performed on samples collected on July 21, 1960. Other samples were collected on June 6, 1959, and Sept. 23, 1959. All samples were composite samples from five to ten Douglas-fir trees, all approximately 40 years old.

The outer bark was removed from the trees with a hatchet and the inner bark was stripped from the trees by hand. The newly formed inner bark was obtained by scraping the inner side of the inner bark with a sharp knife. This white, translucent material was immediately immersed in methanol. Portions of the outer bark and mature inner bark from the sample collected on Sept. 23, 1959, were also placed in methanol.

The method used for the extraction and preliminary separation of the extracts was similar to that used by Mugg (*2*). Within 24 hr. after collection, the methanol was decanted from the sample which was then covered with 90% methanol. After refluxing on a steam bath for 1 hr., the extract was decanted, and the extraction was repeated one more with fresh 90% methanol. The combined extract from each sample was filtered to remove fibrous material, and the methanol was removed under vacuum in a rotary evaporator.

The cloudy aqueous extract was exhaustively extracted with hexane and then filtered through a Celite filter bed. The Celite pad was airdried and extracted with methanol in a Soxhlet extractor.

The filtered water solution after exhaustive extraction with ether, was covered with toluene and placed in a refrigerator in order to prevent decomposition. The yields of the various fractions are reported in Table I.

## ETHER SOLUBLES

In preliminary work an attempt was made to separate the ether solubles by extraction of the ether solution with saturated sodium bisulfite, 5% sodium bicarbonate and 5% sodium hydroxide solutions, successively. After acidification and back-extraction with ether, paper chromatography revealed that essentially no separation had occurred. Therefore, the three fractions were combined, and this separation scheme was not used on other samples.

Paper chromatography of the ether solubles, with 2% acetic acid and the organic phase of *n*-butanol-acetic acid–water (4:1:5) as developing solvents, and using Barton's reagent (*3*) as indicators [diazotized *p*-nitroaniline (*4*) and cinnamaldehyde-hydrochloric acid (*5*)] suggested that *d*-catechin and *l*-epicathechin were present in the newly formed inner bark and mature inner bark ether solubles while the outer bark ether solubles appeared to contain only *d*-dihydroquercetin. The mature inner bark ether solubles also exhibited a very faint spot corresponding to *d*-dihydroquercetin, but the newly formed inner bark ether solubles appeared to contain only *d*-cathechin and *l*-epicathechin.

### *d-Catechin*

A small amount of the newly formed inner bark ether solubles was dissolved in hot water, treated with a small amount of Norite, and after filtration was placed in a refrigerator. After several days the resulting precipitate was recrystallized twice from hot water. The light-tan material sintered at 150°C. and melted at 175 to 176°C. *d*-Catechin sinters at 150°C. and melts at 176 to 177°C. (*6*). The melting point characteristics of the sample did not change when mixed with authentic *d*-cathechin.

The infrared spectrum of this substance was identical to that of an authentic sample of *d*-catechin.

### *l-Epicatechin*

The substance causing the other spot on the chromatograms of the newly formed inner bark ether solubles could not be isolated by crystallization, but it appeared to be identical to an authentic sample of *l*-epicatechin when chromatographed with the solvents recommended by Gage, Douglass, and Wender (*7*). Attempts to isolate this material by heavy-paper chromatography (*8*) were also unsuccessful owing to its poor separation from *d*-catechin.

A solution of the newly formed inner bark ether solubles was streaked on four, 8-in. strips of Whatman No. 1 chromatography paper, which were then developed with the organic phase of *n*-butanol–acetic acid–water (4:1:5). Guide strips were cut from the edges to indicate the location of the two bands, and the bands corresponding to *l*-epicatechin were cut out and extracted with methanol in a micro-Soxhlet extractor.

After evaporation of the methanol to approximately 1 ml., 250 mg. of reagent grade potassium bromide was added, and the slurry was evaporated to dryness under vacuum. The powder was dried overnight in a vacuum oven at 50°C. and was then formed into a pellet for determination of its infrared spectrum. This spectrum was similar to a spectrum of authentic *l*-epicatechin obtained by the same method, but the absorption bands were poorly resolved.

### *d-Dihydroquercetin*

The ether solubles from the outer bark were recrystallized from hot water several times after decolorization with Norite. The melting point of the white crystals was 246 to 248°C., undepressed when mixed with authentic *d*-dihydroquercetin. The acetate was prepared with pyridine and acetic anhydride. Its melting point was 129 to 130°C., undepressed when mixed with the acetate prepared from an authentic sample of *d*-dihydroquercetin.

The existence of a small amount of *d*-dihydroquercetin in the mature inner bark ether solubles was sub-

**Table II. Fractionation of Hexane Solubles from Newly Formed Inner Bark**

| *Fraction* | *Yield, %* | *Description* |
|---|---|---|
| Hexane solubles | 100 | Tan, sticky wax |
| Free acids | 3 | Dark brown wax |
| Unsaponifiables | 5 | Light brown wax |
| Saponifiables | | |
| Ether solubles | 66 | Dark brown sirup |
| Acetone solubles | 27 | Dark brown, amorphous powder |

stantiated by the appearance of a pink color after treating an alcoholic solution of this fraction with zinc and hydrochloric acid. No color was produced by the ether solubles from the newly formed inner bark, but a bright red color was produced by the extract from the outer bark. This color test is very sensitive and has been used for the quantitative determination of *d*-dihydroquercetin (*9*).

In order to evaluate the effect of air-drying, a sample of whole inner bark was collected, but instead of placing this sample in methanol it was allowed to air dry for several months. After grinding in a Wiley mill, the sample was extracted with methanol in a Soxhlet extractor. The methanol extract was diluted with water, and the ether solubles were obtained by exhaustively extracting the aqueous solution remaining after removing the methanol in a rotary evaporator. These ether solubles were compared paper chromatographically with the ether solubles described previously.

By dusting one side of the developed chromatogram with zinc powder and spraying the other side with dilute hydrochloric acid, a red spot appeared corresponding to *d*-dihydroquercetin, but no colors were apparent with *d*-catechin or *l*-epicatechin. It appeared that, relative to the catechins, the amount of *d*-dihydroquercetin in the mature inner bark had increased markedly by air-drying.

## HEXANE SOLUBLES

An attempt was made to separate the hexane solubles by column chromatography on alumina. The column was eluted with petroleum ether, benzene and methanol by the gradient elution method. Although complete separation did not appear to occur, evaporation yielded needle-shaped crystals in several adjacent fractions. They melted at 130 to 132°C. and gave a positive Liebermann-Burchard test for sterols.

The hexane solubles were also characterized by selective extraction and saponification. An ether solution of the hexane solubles was exhaustively extracted with 5% sodium bicarbonate to remove the free acids. The ether was evaporated and the residue was saponified with boiling 10% KOH. After cooling, the solution was extracted with ether to yield a non-saponifiable fraction. After acidification of the alkaline solution, it was extracted with ether to yield a saponifiable, ether-soluble fraction. A substantial amount of dark-brown material remained suspended in the aqueous solution. This material was found to be soluble in acetone. The quantitative results of this characterization are shown in Table II.

**Table III. Estimated Quantities of Sugars in Douglas-Fir Bark**[a]

Percentages based on moisture-free, unextracted bark

| *Sugar* | *Newly formed inner bark* | *Mature inner bark* | *Outer bark* |
|---|---|---|---|
| Arabinose | ... | ... | Trace |
| Glucose | Trace | 1.3 | 0.6 |
| Mannose | ... | ... | ... |
| Xylose | ... | Trace | Trace |
| Sucrose | 16.0 | 2.6 | 0.6 |
| Fructose | Trace | 3.9 | 0.6 |
| Raffinose | ... | ... | ... |

[a] Sample collected Sept. 23, 1959.

**Table IV. Estimated Quantities of Sugars in the Newly Formed Inner Bark of Douglas-Fir**

Percentages based on moisture-free, unextracted bark

| *Date sample collected* | *Sucrose* | *Glucose* | *Fructose* | *Raffinose* |
|---|---|---|---|---|
| June 6, 1959 | Present[a] | Present[a] | Present[a] | Present[a] |
| July 21, 1960 | 27.0 | 0.7 | 0.7 | 0.5 |
| Sept. 23, 1959 | 16.0 | Trace | Trace | Trace |

[a] Quantities not estimated.

## METHANOL SOLUBLES

Paper chromatography indicated that the methanol-soluble fraction was identical to the water-soluble, phenolic substances. It was also found that this fraction contained a considerable amount of mineral matter. This fraction was not further investigated.

## WATER SOLUBLES

Preferential solvent extraction, countercurrent extraction, and column chromatography were used in attempts to separate the polyphenols in this fraction. With none of these methods could a substantial amount of homogeneous, phenolic material be obtained. Infrared spectra of the ethyl acetate-extractable materials indicated that only the extract from the outer bark contained substances with carbonyl groups. However, paper chromatography revealed that the ethyl acetate-solubles from the outer bark contained a small amount of *d*-dihydroquercetin, which would account for the carbonyl absorption band in the infrared spectrum.

### *Sugars*

The water solubles were examined paper chromatographically using *n*-butyl acetate–pyridine–ethanol–water (8:2:2:1) as the developing solvent and *p*-anisidine hydrochloride (*10*) and α-naphthol–phosphoric acid (*11*) as indicators. The quantities were estimated by visual comparison with known concentrations of standard sugars. The sugars and their approximate amounts are reported in Table III.

The sugars from the newly formed inner bark collected on July 21, 1960, were also estimated quantitatively. The sugars present in the sample collected on June 6, 1959, were determined, but the quantities not estimated. These results are given in Table IV.

### *Ion-Exchange Separation*

The water solubles were next separated by ion-exchange chromatography. A portion of the water solubles in aqueous solution was treated with a saturated lead acetate solution to precipitate the polyphenolic materials. The excess lead was removed by precipitation with hydrogen sulfide.

After filtration, the solution was added to a column of Dowex 50-x4 cation-exchange resin in the hydrogen ion form. The column was washed with distilled water until a positive Molisch test for carbohydrates was not obtained. The combined effluent was then passed

Table V. Amino Acids in the Newly Formed Inner Bark

| Amino Acid | Amount[a] |
|---|---|
| Aspartic acid | ++ |
| Glutamic acid | ++ |
| Serine | ++ |
| Proline | + |
| Glycine | + |
| Alanine | + |
| Threonine | + |
| Valine | + |
| Leucine | + |
| Isoleucine | + |
| Phenylalanine | Trace[b] |
| Tyrosine | Trace[b] |

[a] ++ = >0.01%; + = <0.1%; both based on moisture-free, unextracted bark.
[b] Presence of these compounds doubtful.

through a column of Dowex 1-x4 anion exchange resin in the hydroxyl ion form. This column was also washed with distilled water until a positive Molisch test was not obtained on the effluent. The total effluent was evaporated under vacuum to a small volume to yield a neutral fraction.

The cation exchange column was eluted with 2*N* $NH_4OH$ until the effluent was distinctly basic. The column was then washed with distilled water until the effluent was neutral. The total effluent was combined and evaporated to dryness in a rotary evaporator to yield a water-soluble, base fraction.

The anion-exchange column was eluted with 2*N* HCl in a similar manner to yield a water-soluble, acid fraction. The base fraction was a tan powder while the acid fraction was a light-gray, deliquescent solid.

### *Amino Acids*

The amino acids in the base fraction were determined by paper chromatography, using 80% phenol and the organic phase of *n*-butanol–acetic acid–water (25:6:25) as developing solvents, and 0.2% ninhydrin in *n*-butanol saturated with water as the indicator. Since the concentration of each of the amino acids was approximately the same, standards of appropriate concentrations were chosen so that it could be determined whether the amino acids were present in quantities less than or greater than an arbitrary amount, 0.01%, based on the moisture-free, unextracted bark. The amino acids detected and their concentration limits are given in Table V.

### *Water-soluble Acids*

The water-soluble acid fraction was examined paper chromatographically for uronic acids. No evidence for any sugar acid could be found although a bromophenol blue spray (*12*) indicated that several acids were present. The $R_f$'s and color reactions of these acids indicated that they were neither simple aliphatic acids nor any of the acids which occur in the Krebs tricarboxylic acid cycle.

When Cartwright's (*13*) hydroaromatic acid spray was used, several spots appeared. This spray is assumed to be specific for hydroaromatic acids. Ethyl acetate–acetic acid–water (3:1:3) caused separation into two strong spots and six very weak spots. The two strong spots corresponded to shikimic and quinic acids, respectively. A chromatogram using this solvent is illustrated in Fig. 1.

The acid mixture was also chromatographed using

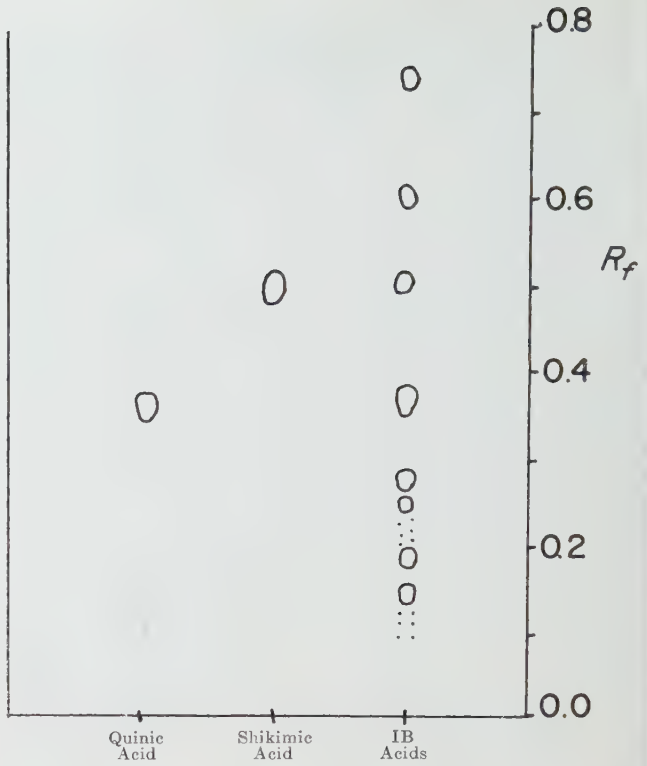


*Fig. 1. Chromatogram of hydroaromatic acids*

Developer: ethyl acetate–acetic acid–water (3:1:3)
Indicator: Cartwright's hydroaromatic reagent

*n*-butanol–pyridine–water (10:3:3), phenol-water (3:1) with 0.9% formic acid and *n*-butanol–acetic acid–water (4:1:5). With each of these solvents the two strongest spots were identical to quinic and shikimic acids, respectively. Owing to the small amounts of the substances responsible for the six weak spots, these were not evident on all chromatograms. The amounts of quinic and shikimic acids were estimated as 0.1% and 0.03%, respectively, based on the moisture-free, unextracted bark.

Hathway (*14*) found that the phenol–water–formic acid solvent separated shikimic acid from 5-dehydroshikimic acid, but only the two spots corresponding to shikimic and quinic acids were evident when this solvent was used.

Since the phosphate esters of the hydroaromatic acids are presumed to take part in the biosynthetic pathways of bacteria, a chromatogram of the acids was treated with the phosphate spray of Hanes and Isherwood (*15*). No spots appeared when this spray was used.

### *Water-soluble neutrals*

When the water-soluble neutrals were chromatographed with 2% acetic acid and *n*-butanol–acetic acid–water (4:1:5), very strong spots appeared after the chromatograms were sprayed with the *p*-anisidine–hydrochloric acid spray. All of these spots corresponded to the previously determined sugars. In addition, another spot was found which had $R_f$'s of 0.82 and 0.88 in *n*-butanol–acetic acid–water (4:1:5) and 2% acetic acid, respectively. This spot did not give positive results with either Barton's reagent or diazotized *p*-nitroaniline, both of which are general phenol indicators. This spot was orange with both 2,4-dinitrophenylhydrazine (*16*) and Wiesner's reagent (*17*).

Since vanillin and coniferaldehyde, both of which are

Table VI. Paper Chromatography of Wiesner-Positive Materials

| Substance | $R_f \times 100$ BAW[a] | $R_f \times 100$ 2% HAc[b] | Color tests UV[c] | Color tests Wiesner reagent |
|---|---|---|---|---|
| Unknown | 82 | 88 | Dark blue | Orange |
| Vanillin | 91 | 67 | Dark blue | Peach |
| Coniferaldehyde | 89 | 47 | Light blue | Cerise |

[a] Organic phase of *n*-butanol–acetic acid–water (4:1:5).
[b] 2% acetic acid.
[c] Color under ultraviolet light.

positive to Wiesner's reagent, have previously been reported in Douglas-fir bark (*5*), these two compounds were run in parallel to the unknown. The results of these chromatograms are reported in Table VI.

Since the Wiesner-positive substance was soluble in ether, it was removed from the sugars in the aqueous solution by extraction. The material appeared to be chromatographically pure after the extraction.

Although the solubility of this substance in ether indicated that it was not a glycoside, an aqueous solution of the compound was treated with emulsion for 24 hr. at 33°C. No change was evident by paper chromatographic analysis.

The compound was decomposed by treating with 1*N* HCl at 105°C. for 3 hr. in a sealed glass tube. Results of paper chromatography of the hydrolyzate were very difficult to interpret, owing to their complexity. Very weak spots were found corresponding to glucose and arabinose, but the results were far from conclusive. Most of the hydrolyzate either did not move or streaked the entire length of the chromatograms with each of the solvents used. A color test for this material could not be obtained, but it fluoresced under ultraviolet light.

Owing to the paucity of the Wiesner-positive material, it could not be recrystallized. The ultraviolet spectrum of the substance exhibited maximum absorption at 280 mμ and minimum absorption at 244 mμ. No shift was observed in alkaline solution.

It is interesting that no evidence of this substance could be found in any of the other fractions of the total methanol extract.

### *Marcs*

The ash and lignin contents of the newly formed inner bark marc were compared with those of the mature inner bark and outer bark marcs by means of TAPPI Standards T 15 m-58 and T 13 m-54, respectively. The results of these determinations are given in Table VII.

Table VII. Analysis of Extracted Bark
Percentages based on moisture-free, extracted bark

| Sample | Lignin | Ash |
|---|---|---|
| Newly formed inner bark | 18.79 | 5.19 |
| Mature inner bark | 31.74 | 0.69 |
| Outer bark | 79.35 | 0.21 |

## DISCUSSION

The predominance of *d*-catechin and *l*-epicatechin in the ether-soluble fraction from the newly formed inner bark would seem to indicate that the catechins are precursors of components of either the bark or wood. This agrees with the generally accepted theory that many of the high-molecular-weight polyphenols; namely, tannins, phlobaphenes, and possibly bark lignins, are polymers of catechins and related substances. The absence of catechins in the nonliving portion of the bark (outer bark) further substantiates this theory.

Although Weinges (*19*) recently reported the presence of the two catechins in fresh, airdry Douglas-fir bark, it seemed strange that previous investigations of this bark had not revealed their presence. In this work is was found that prolonged air-drying of the inner bark appeared to increase the amount of *d*-dihydroquercetin and/or decrease the amounts of the catechins. However, the quantities of the flavonoids were only shown to change in relation to each other.

The presence of sucrose in the extract from the newly formed inner bark was not unexpected as this sugar has been shown to be present in the cambial zone of many trees. Kremers (*19*) has stated that sucrose is the principal soluble reserve food supply.

Glucose and fructose are also quite commonly found in the cambial zone of trees. Glucose has been shown to be the precursor of lignin (*20*) and flavonoids (*21*), and it is obviously a precursor of cellulose. It might be assumed that glucose is the principal precursor for all of the wood and bark components.

The role of raffinose is somewhat debatable, but Zimmermann (*22*) has postulated that galactosides may play a part in the transfer of photosynthates from the sieve tubes. It is interesting that raffinose was not found in the sample collected in September. Grillos (*23*) has shown that cell division in Douglas-fir ceases in August, thus raffinose would not necessarily be present in the cambium between August and the commencement of growth, in the spring.

The lack of conclusive evidence for the presence of phenylalanine and tyrosine is a paradox, since these aromatic amino acids are presumed to play an important role in the formation of lignin. However, it is not possible to conclude from chromatographic data that these compounds are absent, and it is possible that they may be highly transitory.

The apparent absence of methionine also presents a problem, since this amino acid is presumed to be responsible for the methylation of phenols; e.g., methylation of catechol nuclei to guaiacyl nuclei. It is interesting that Mugg (*2*) did not find this amino acid in aspenwood.

It would seem likely that the majority of the amino acids are present for the synthesis of proteins (enzymes) rather than the synthesis of lignin or cellular components.

The presence of the hydroaromatic shikimic and quinic acids is quite significant, especially since these substances were apparently not present in the cambial zone of aspen (*2, 24*). The hydroaromatic acids are the intermediates between carbohydrates and aromatic compounds (*25*), and they have been shown to be precursors of lignin (*26*) and flavonoids (*21*). Whether the presence of the hydroaromatic compounds in the cambial zone indicates that aromatization and dimerization to flavonoids occurs there is debatable, since Hergert and Goldschmid (*28*) have presented evidence that synthesis of the flavonoid *d*-dihydroquercetin occurs in the needles of conifers. It may be significant that phloroglucinol, which may be another precursor of flavoniods (*21*), is aslo present in the cambial zone of Douglas-fir (*5*).

Of particular interest was the chromatographic indication of six unidentified, apparently hydroaromatic

substances. These materials were present in very small amounts, and it is possible that they were either 5-dehydro or phosphate derivatives of quinic or shikimic acids. However, no phosphates could be detected by a color test, and no chromatographic evidence for the 5-dehydro compounds was found.

Since the unidentified, Wiesner-positive material was evidently not a phenol, it was thought that it may have been a glycoside. Emulsion had no effect on the compound, but hot, dilute mineral acid completely destroyed it. No compounds were definitely identified in the hydrolyzate, but meager evidence was obtained for glucose and arabinose.

This Weisner-positive material was easily extracted from an aqueous solution with ether; therefore a reexamination of the ether-soluble fraction was made. No Wiesner-positive substance could be found in this or any other fraction. Thus, the possibility exists that this substance was an artifact, possibly derived from the sugars.

The sulfuric acid "lignin" content of the bark increased from the newly formed inner bark to the outer bark, as would be expected. However, the relatively high amount (18.79%) of "lignin" in the newly formed inner bark was unexpected. This was undoubtedly due to the fact that proteins and other nonlignin materials were insoluble in the sulfuric acid. In agreement with these results, Stewart (*27*) found 21% lignin in the cambial zone of *Eucalyptus regnans* by the sulfuric acid method, but by indirect means he showed that the true lignin content was less than 1%.

The relatively high ash content of the newly formed inner bark is indicative of the amount of mineral matter necessary for biochemical activity in the cambial zone. The much lower ash content of the outer bark shows that the generally higher ash content of bark, as compared to wood, is not due to wind-blown dust, etc.

## CONCLUSION

The relatively homogeneous nature of the ether solubles from the newly formed inner bark of Douglas-fir, as compared to those of aspen (*24*), may be significant. Although no methoxylated aromatic compounds were identified in this fraction, Hergert (*5*) has indicated that vanillin, coniferaldehyde, and ferulic acid are present in Douglas-fir bark. These compounds are probably involved in wood lignin synthesis. However, the origin of the methoxyl groups in Douglas-fir bark tannin (*29*) and lignin (*1*) remains unknown. It is conceivable that the unidentified, Wiesner-positive material found in the newly formed inner bark of Douglas-fir is a methoxylated precursor of either wood or bark lignin.

The alkaline nitrobenzene oxidation products of Douglas-fir bark lignins, vanillin and protocatechualdehyde (*1*), would seem to indicate that the bark lignins are copolymers of compounds containing both catechol and guaiacyl nuclei. In addition to vanillin and protocatechualdehyde, phloroglucinol was detected in the oxidation products of white fir bark lignins (*30*). These products may indicate that the bark lignins are copolymers of guaiacyl compounds and catechins, or related substances. It is interesting that white fir bark has also been shown to contain substantial amounts of catechins (*6*).

If the above conclusions are correct, the predominance of the catechins in the ether solubles of Douglas-fir newly formed inner bark would be justified. Whether the catechins are synthesized in the cambial zone is problematical, especially in the light of the evidence presented by Hergert and Goldschmid (*28*). They indicated that the synthesis of another flavonoid, *d*-dihydroquercetin, probably occurs in the needles (leaves) of conifers.

The lack of conclusive evidence for the presence of the amino acids tyrosine, phenylalanine, and methionine in the newly formed inner bark of Douglas-fir would seem to be contrary to the present knowledge of lignin biosynthesis (*31*). It is believed that the absence of these compounds only strengthens the hypothesis that the amino acids are primarily responsible for protein (enzyme) synthesis, rather than the synthesis of structural components of the tree.

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# Influence of Surface Roughness Upon the Measurement of Tritium Radioactivity in Paper and Other Sheets

O. SEPALL and S. G. MASON

**The effect of surface roughness on the measured radioactivity of paper, cellophane, and starch sheets containing tritiated hydroxyl groups was studied theoretically and experimentally. Calculated counting rates for various models suggest that the important roughness parameter is the ratio of depth $Z$ to width $W$ of surface asperities. Corresponding to a fixed degree of tritiation, values of $Z/W > 1$ cause the solid counting rate to be greater, and $Z/W < 1$ to be less than that of a flat sample. The bulk density of the sample is not important. Measurements on various materials provided general confirmation of the theory. It is concluded that solid counting is a valid procedure for measuring the degree of tritiation in paper and similar sheets.**

## I. INTRODUCTION

In an investigation (*1*, *2*) of hydrogen exchange between cellulose and tritiated water (HTO), there was evidence that tritium radioactivity in the solid samples was influenced by surface roughness. The effect of roughness upon the radioactivity of solid samples appears not to have been studied although the factor is clearly important when soft beta emitters are used. With cottonseed oil triglyceride, the counting rate for tritium was reported to be doubled by solidfying the sample, presumably as a result of the accompanying increase in surface area (*3*).

This paper describes an investigation of the influence of the topography of tritiated cellulose samples upon their radioactivity. Counting rates were calculated for various models and the effect of the two principal variables, sample bulk density and shape of superficial irregularities, was considered. The calculations were compared with measurements on a variety of samples. It was found that a parameter describing the cross-sectional shape of the irregularity was related to the counting rate.

### NOMENCLATURE

| | | |
|---|---|---|
| $A$ | = | surface area, cm.$^2$ |
| $C = N/n$ | = | fraction of particles emitted which are counted |
| $D$ | = | deviation in count rate from that of ideal flat sample |
| $K$ | = | rate of beta particle emission per unit volume, cm.$^{-3}$ sec.$^{-1}$ |
| $n$ | = | total rate of beta particle emission, sec.$^{-1}$ |
| $N$ | = | total count rate, sec.$^{-1}$ |
| $r, \theta, \phi$ | = | standard spherical polar coordinates |
| $R$ | = | maximum beta particle range, cm. |
| $V$ | = | volume, cm.$^{-3}$ |
| $W, Z$ | = | width and thickness of rectangular fiber, cm. |
| $x, y$ | = | vertical distances in Model II, Fig. 2, cm. |
| $\alpha$ | = | solid angle (steradians) |
| $\gamma$ | = | angle in Model I, Fig. 2. |
| $\mu$ | = | absorption coefficient of beta particles, cm.$^{-1}$; also used for linear distance in microns, $10^{-4}$ cm. |
| $\rho$ | = | radius of sphere, cm. |
| $\sigma$ | = | flux of beta particles, cm.$^{-2}$ sec.$^{-1}$ |

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## II. EXPERIMENTAL

### *Methods*

The influence of surface characteristics upon the tritium radioactivity of cellulose sheets was estimated from comparative measurements of the exchange of hydrogen isotopes between cellulose and water as previously described (*2*). In one method, the exchange ($A_1$ % of hydroxyl reacted) of deuterium with heavy water ($D_2O$) was determined gravimetrically (*2*); this method is absolute. In the other, the exchange ($A_2$) of tritium with tritiated water (HTO) was measured by a solid counting technique (*2*). The isotope effect is different in each method but it did not affect the comparison because the radioactivity measurements were calibrated by assuming $A_1 = A_2$ for a smooth cellophane sample. The difference in the results ($A_1 - A_2$) with other samples was therefore ascribed to surface irregularities of the solid samples.

### *Materials*

The following materials were used:

*Cellophane:* air-dried film containing no additives (du Pont of Canada, Ltd.).

*Wood cellulose:* softwood acetate grade woodpulp (alpha-cellulose = 96% by TAPPI Standard T 203 m-58).

*Cotton linters:* dewaxed by alcohol/benzene extraction.

*Bacterial cellulose:* kindly supplied by Dr. R. J. Colvin of the National Research Council of Canada.

*Amylose:* commercial grade extracted from potato starch (Stein Hall and Co.).

*Wood pulp:* paper grade bleached softwood sulfite (Anglo Paper Products, Ltd.).

The samples were prepared in sheets of about 3 mg. per sq. cm. Amylose was dispersed in water at 160°C. and a film deposited on a polished chromium-plated surface by drying at room temperature. Handsheets were prepared from the fibrous materials according to TAPPI Standard T 205 m-58. Cotton linters required a preliminary beating in a PFI* laboratory mill before a satisfactory sheet could be prepared. Cellophane was used as received.

It was of interest to investigate specimens in which the roughness could be varied regularly over a wide range. This was done by using samples of paper prepared from pulps beaten to different extents with a

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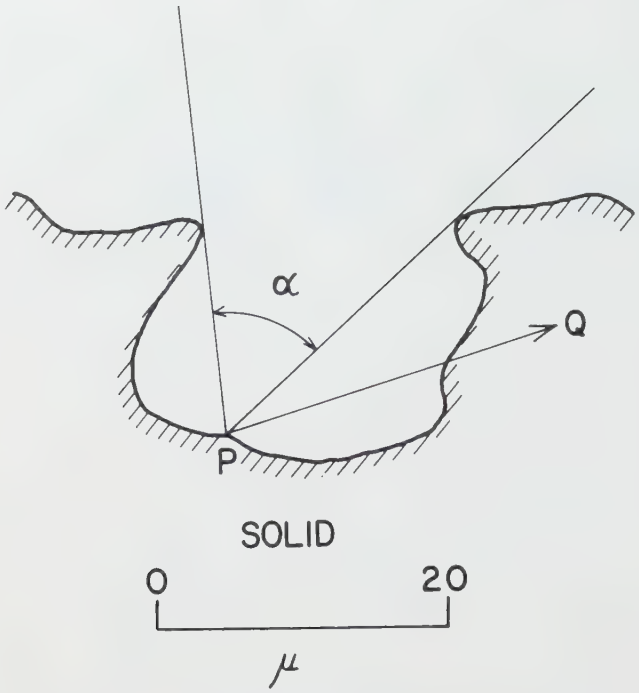


*Fig. 1. Sectional view of assumed cavity in cellulose*

Valley laboratory mill. The exchange properties were assumed to be unaffected by beating since the related property of sorptive capacity for water has been shown to be unchanged after such treatment (*4*).

Electronmicrographs of the sheet surfaces were prepared from specimens metallized with 60/90 gold/palladium, using a scanning electron microscope (*5*).

## III. THEORETICAL

### *General Considerations*

The calculations were made only for tritium in infinitely thick sheets of cellulose and starch counted under the conditions of the measurements described elsewhere (*2*), i.e., using methane gas at 90 mm. Hg in the proportional region. Because of its complexity a more general treatment was not attempted.

To calculate counting rates for model solids required (a) a knowledge of the conditions under which a beta particle is counted, and (b) an estimate of the surface flux. The assumptions and approximations made are discussed below.

### *Assumptions*

*Conditions for a beta particle to register a count.* To register a count a beta particle must have sufficient energy and a suitable path in the counter gas to generate the required least number of ion pairs. The manner in which the surface irregularities determine the conditions is as follows.

Surface cavities (Fig. 1) approximately 20 $\mu$ wide, corresponding to the larger voids in the samples studied, are assumed. As will be evident later, the dimensions are not critical and cavities much smaller than 20 $\mu$ may be neglected. It is assumed that the first 20 $\mu$ of the path of an emitted particle, as from $P$ in Fig. 1, is rectilinear because deviations in the path in gas are small except at very low particle energy. The additional velocity imparted to the particle by the electric field of the counter is small and may be neglected. Thus, the particles emitted within the solid angle $\alpha$ subtended at $P$ by the opening of the cavity escape directly into the counter gas. The particles emitted outside the angle $\alpha$, as along $PQ$, collide with a cellulose surface and are either back-scattered or absorbed. The thickness of cellulose at the wall is assumed to be of wood fiber dimensions (about 20 $\mu$) and therefore greater than the maximum particle range, $R$, of 5 $\mu$ (*6*), corresponding to 0.8 mg. per sq. cm. of solid cellulose; saturation back-scattering thus occurs, which was estimated to be 10% for beta particles impinging on solid substances of similar atomic number (*7*). The remaining 90% of the particles striking the wall are assumed to be absorbed, neglecting those which can penetrate completely through solid near the top edges of the cavity.

Whether particles emitted along different paths are counted depends upon the extent of ionization created. The particles which traverse a cavity in the sample surface and are subsequently absorbed by cellulose, may produce ion pairs along the path in the gas. The number of primary ion pairs formed is less than the number of collisions between beta particles and gas molecules.

The mean free path of an electron in methane at 90 mm. Hg is about 10 $\mu$ (*8*). A beta particle traveling 20 $\mu$ in the gas collides on the average with two gas molecules and produces fewer than two primary ion pairs. This ionization is assumed to be negligible. The assumption is usually valid because counters are often operated at low sensititivity to improve stability. For example, in the present measurements, it is estimated from the variation of plateau counting rates with discriminator bias that at least 10% of the total beta particles are not registered. Since the uncounted particles are in the low range of the energy spectrum to at least 900 ev. (*9*), more than 30 ions pairs were required for a count, assuming an energy loss of about 30 ev. per ion pair as reported for alpha particles (*10*).

Thus, to summarize, it is assumed (a) that a particle emitted from a surface can be counted if it is included within a solid angle unobstructed by adjacent surfaces, and (b) of the particles striking cellulose 10% undergo back-scattering and may then be counted if re-emitted in angle $\alpha$.

For a flat surface $\alpha = 2\pi$ and all emitted particles having sufficient energy are counted. At any point on an irregular surface, the fraction counted is therefore $\alpha/2\pi$ assuming that particles are emitted with a random angular distribution (this is believed to be approximately correct). The nature of the angular distribution is discussed in Section V and shown to depend upon the path of the particle in the solid.

Cavities larger than 20 $\mu$ were not considered because, as will be seen later, they were not common in the samples. It may be noted, however, that ionization in the cavity will cause larger cavities and higher gas pressures to increase the counting rate.

If the dimensions of the cavity in Fig. 1 are small compared with the particle range $R$ (5 $\mu$), the particles emitted outside the angle $\alpha$ will not be absorbed when the thickness of cellulose is much smaller than $R$. Under these conditions, $(\alpha/2\pi)$ approaches 1 and the effect of the cavity upon $\alpha$ may be neglected.

*Total number of particles emitted.* The number of particles emitted by a solid from the area $dA$ is $\sigma_n dA$

**Table I. Effect of Radius of Sphere upon $\sigma_n$**

| *Radius of sphere* | $\sigma_n''/\sigma_n'$ |
|---|---|
| $4R$ | 0.95 |
| $2R$ | 0.90 |
| $R$ | 0.80 |

$\sigma_n''$ = flux from sphere.
$\sigma_n'$ = flux from flat solid.

where $\sigma_n$ is the surface flux of particles emitted at $dA$ with components of velocities normal to the surface. The total rate of particle emission by the solid is therefore:

$$n = \int_{A_S} \sigma_n dA \qquad (1)$$

where $A_S$ is the surface area of the sample. The use of equation (1) requires a knowledge of the effect of curvatures upon $\sigma_n$. This was estimated by calculating $\sigma_n$ for spheres of tritiated cellulose of different radii $\rho$.

The particles emitted from a solid and originating from volume element $dV$ may be written as $Kf(r, \phi, \theta)dV$, where $K$ is the total number of particles emitted per unit volume; $f(r, \phi, \theta)$ represents the fraction of particles reaching the surface, and depends upon the distance to the surface and the attenuation of the radiation. Thus:

$$n = \int_{V_S} Kf(r,\phi,\theta)dV \qquad (2)$$

where $V_S$ is the volume of the surface layer which extends to a mass depth equal to the maximum particle range, 5 $\mu$ in cellulose and starch.

The flux $\sigma_n$ was calculated for a flat solid and for spheres of radii $\rho = R$, $2R$ and $4R$, by assuming that particles are emitted randomly from every point in the solid and are absorbed exponentially with distance traveled. Since exponential absorption is an approximation the calculations indicate only the order of magnitude, which is adequate for the present purpose.

The calculated values of $\sigma_n$ (Table I) show little variation when $\rho > 4R$. When $\rho < 4R$, $\sigma_n$ decreases with $\rho$.

At a large surface with irregularities of $\rho < 4R$, $\sigma_n$ does not necessarily change in the same way as with isolated spheres. Differences in $\sigma_n$ at adjacent concave (curvature $> 0$) and convex (curvature $< 0$) regions also tend to cancel out. Value of $\sigma_n$ was not calculated in such cases because of the difficulty of integrating equation (2). However, it is evident that those surface irregularities which are small compared with $R$ emit only a small fraction of the total radioactivity and may therefore be neglected.

It is thus probable that the average flux at any irregular surface is the same as for a flat solid. The radii of curvature in all the models studied was greater than $2R$, so that the total number of emitted particles was $\sigma_n' A_S$ where $\sigma_n'$ is the flux from a flat solid.

When particles impinge on the surface, the flux becomes $(\sigma_n' + \sigma_{nB})$ where $\sigma_{nB}$ is the flux corresponding to back-scattering, i.e., 10% of the impinging particles.

### *Calculations of Counting Rates for Models*

From the foregoing considerations, the counting rate for area $dA$ is:

$$dN = C(\alpha/2\pi)\,(\sigma_n' + \sigma_{nB})\,dA \qquad (3)$$

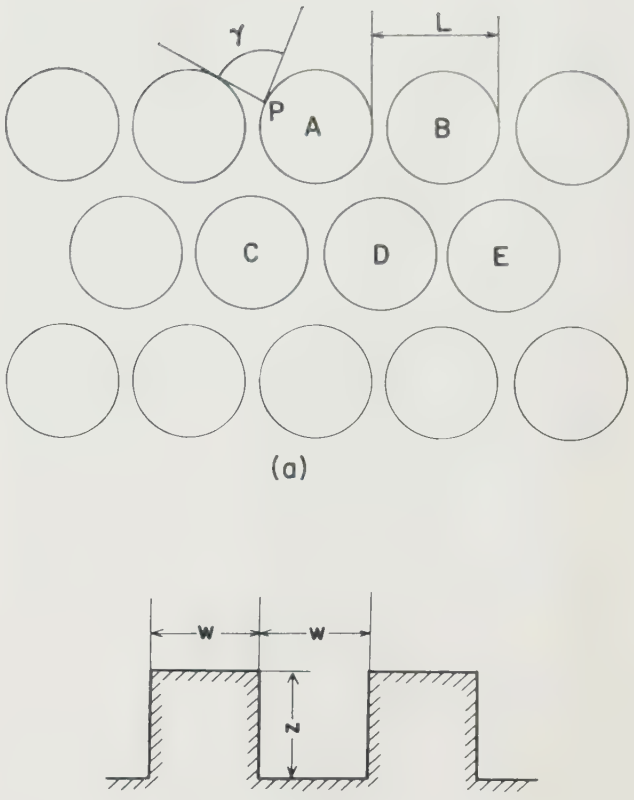


*Fig. 2 Sectional view of models for paper samples*
(a) Model I (b) Model II

where $C$ is a factor ($< 1$) representing the fraction of particles counted, and depends upon the counter sensitivity. The remaining terms give the number of particles which are capable of registering a count assuming a random angular distribution of velocities for the particles emitted at the surface. The total counting rate $N$ was calculated either in closed form or by numerical integration of equation (3) for a number of models.

### *Description of Models*

The samples studied included smooth films and papers made from fibrous materials. The film samples are represented by a flat solid and the fibrous materials by simple models consisting of long straight fibers of circular and rectangular cross-sectional shapes and arranged in parallel rows. In Model I, shown in sectional view in Fig. 2a, the cylindrical fibers are uniformly spaced at intervals corresponding to the bulk density desired. In Model II, Fig. 2b, the ratio of fiber width-to-thickness ($Z/W$) for the rectangular section is varied to represent the flattened shape of wood fibers.

## IV. RESULTS AND DISCUSSION

The calculated and measured counting rates are given in Tables II and III, respectively.

The calculated values in Table II are expressed in terms of the deviation $D$ given by:

$$D_{\text{calcd.}} = \left(\frac{N' - N}{N} \times 100\right) \qquad (4)$$

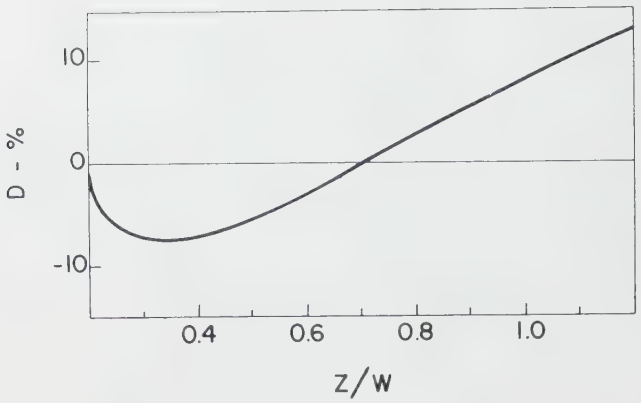


*Fig. 3. Effect of (Z/W) upon D with Model II*

where $N$ is the counting rate for the model and $N'$ is the rate for a flat solid of the same overall dimensions. The measured values are expressed as the corresponding deviation:

$$D_{meas.} = \left(\frac{A_1 - A_2}{A_1}\right) \times 100 \qquad (5)$$

Cellophane was assumed to be flat so that $A_1 = A_2$ and hence $D = 0$.

The calculations indicated that both positive and negative deviations could occur. With Model I, $D_{calcd.} > 0$ and independent of the bulk density. In Model II (Fig. 3) with deep narrow depressions, i.e., for $(Z/W) > 1.0$, $D > 0$; when $(Z/W) < 1.0$, $D < 0$. It should be noted that $D_{calcd.} = 0$ for both $(Z/W) = 0$ and 1.

The measured and calculated $D$'s were in general agreement. With amylose film, $D$ was small (2%), as would be expected since the sample was smooth as cellophane. These films were generally free of irregularities larger than 1 $\mu$. Smaller irregularities were inconsequential for reasons discussed in Section III.

Negative deviations were found with bacterial cellulose and papers prepared from beaten pulps. Figure 4a shows that bacterial cellulose consisted of fine strands of less than 1 $\mu$ thickness and the surface, although irregular and undulating, was without deep depressions. In the sample of beaten pulp, Fig. 4b, the fibers were flattened and the superficial cavities appeared to have little depth in comparison to their width, corresponding to $(Z/W) < 1$ and a negative $D$ for Model II.

$D$ was positive for cotton linters, which is shown by Fig. 4c to be highly porous. The fibers appeared to be stiff, uncollapsed, and poorly bonded to each other so that extensive voids (indicated by the low bulk density, Table III) were present in the sheet. Although a value of $(Z/W)$ cannot be realistically assigned to the sample it is greater than unity, so that $D$ is expected to be positive as with Model II. The unbeaten wood cellulose paper, Fig. 4d, had fewer voids than cotton linters, but cavities in the surface were more extensive than in the corresponding beaten sample. The appearance thus corresponds to a value of $(Z/W)$ intermediate between cotton linters and beaten pulps. The value of $D$ for unbeaten wood cellulose was also intermediate and corresponds to Model II with $Z/W = 1.0$. The results thus appear to depend upon the ratio of depth-to-width of the superficial irregularities. It is also of interest to note from Fig. 3 that irregularities having a distribution of $Z/W$ between 0 and 2.0, because of the averaging of positive and negative $D$'s, could give the same result as $Z/W = 1.0$. This could account for the results with the unbeaten wood cellulose.

**Table II. Calculated Deviations in Counting Rates**

| *Model* | *Description* | $D_{calcd.}$, % |
|---|---|---|
| Flat solid | | 0 |
| I | Bulk density = 0.87 g./cc. | +7 |
| I | " " = 0.75 g./cc. | +6 |
| II | $Z/W$ = 1.0; bulk density = 0.75 g./cc. | 0 |
| II | $Z/W$ = 1.5; " " " " | +7 |
| II | $Z/W$ = 0.3; " " " " | −7 |

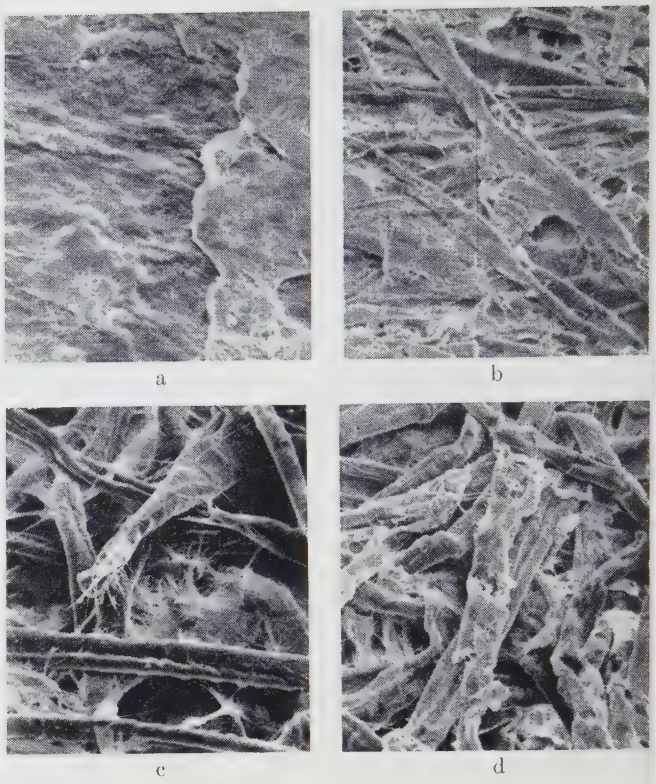


*Fig. 4. Electronmicrographs of sheet surfaces*

(a) Bacterial cellulose
(b) Beaten woodpulp (pulp was beaten to 100 C.S.f.—Table III)
(c) Cotton linters
(d) Unbeaten wood cellulose

The variation of bulk density from 0.75 to 0.85 g. per cc. with Model I, and from 0.67 to 0.91 g. per cc. with the wood pulp sample, had no effect upon the values of $D$. With other samples, even larger variations were apparently unrelated to $D$ so it is concluded that bulk density is unimportant.

**Table III. Measured Deviations in Counting Rates on Cellulose Samples**

| *Sample* | *Bulk density, g./cc.* | $D_{meas.}$, % |
|---|---|---|
| Cellophane | 1.5 | 0 |
| Amylose | ... | +2 |
| Wood cellulose | 0.42 | 0 |
| Bacterial cellulose | 1.10 | −7 |
| Cotton linters | 0.29 | +10 |
| Wood cellulose (beaten to 225 C.S.f.[a]) | 0.67 | −2.5 |
| Wood cellulose (beaten to 100 C.S.f.) | 0.72 | −2.5 |
| Wood pulp (unbeaten) | 0.67 | 0 |
| Wood pulp (beaten to 516 C.S.f.) | 0.80 | 0 |
| Wood pulp (beaten to 329 C.S.f.) | 0.91 | 0 |
| Wood pulp (beaten to 131 C.S.f.) | 1.00 | −3.5 |

[a] Canadian Standard freeness.

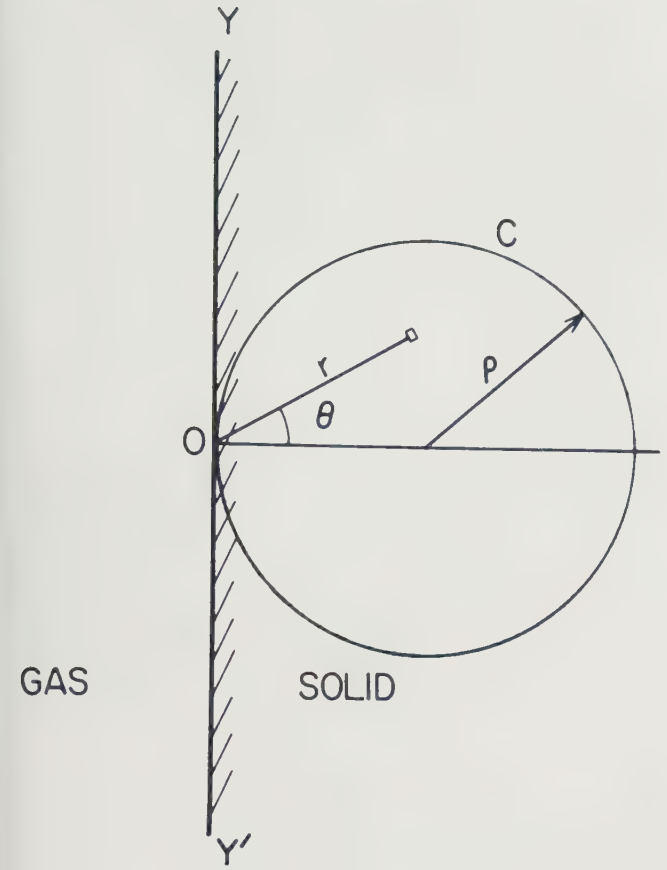


*Fig. 5. Coordinate system for calculations of $\sigma_n$ for flat and spherical surfaces*

(for details see text)

There are, of course, serious limitations to these calculations. For example, randomly arranged fibers were not considered although they may considerably influence the angle $\alpha$. It must also be emphasized that the results are only approximate because of the many simplifying assumptions. However, they are useful because they lead to several interesting predictions. Thus, the effect of superficial irregularities is predicted to be greater for (1) higher counter gas pressures, (2) larger superficial cavities, (3) more sensitive counters, (4) substances composed of elements of higher atomic number for which back-scattering is greater, and (5) certain patterns of surface irregularities such as deep narrow cracks.

In spite of surface roughness, the measurement of tritium radioactivity in various cellulose samples was usually accurate to ±5%. This is considerably better than the results previously obtained by solid counting techniques on other materials (*3*). Tritium tracer studies in cellulose and related substances therefore merit more widespread use.

## V. DETAILS OF CALCULATIONS

### $\sigma_n$ for a Flat Solid

Let point $O$ in the flat surface $YY'$ shown in Fig. 5 be the origin of coordinates. The flux $\sigma_n$ at $O$ originates from a hemisphere of radius $R$ and center $O$. If $K$ particles per cc. per sec. are emitted in the solid, then $\sigma_n$ for the volume element $dV$ is given by:

$$d\sigma_n = K\, e^{-\mu r}\, \frac{1}{2}\, \frac{1}{4\pi r^2}\, r^2 \sin\theta\, d\theta\, d\phi\, dr \qquad (6)$$

Here $e^{-\mu r}$ is the attenuation term for exponential absorption, $\mu$ is the absorption coefficient for beta particles from tritium in cellulose, $^1/_{4\pi}r^2$ is the fraction of the total solid angle subtended at the volume element by unit surface area at $O$ of the sphere of radius $r$. The fraction $^1/_2$ arises from the assumption of random particle paths; this implies that $^1/_2$ of the particles reaching $O$ have a component of velocity along $\theta = \pi$, while the remainder have a component along $\theta = 0$.

If rectilinear paths had been assumed, the term $(^1/_{8\pi}r^2)$ would have been replaced by $(\cos\theta/4\pi r^2)$, i.e., the fraction of the total solid angle subtended at the the volume element by the projection of unit surface area of the solid at $O$, and the same result obtained.

Integration of equation (6) over the hemisphere yields:

$$\sigma_n = \frac{K}{4\mu}(1 - e^{-\mu R}) \qquad (7)$$

The attenuation at distance $R$ is great, so that $e^{-\mu R}$ is small compared to 1.0 and may be neglected. Equation (7) thus reduces to:

$$\sigma_n = \frac{K}{4\mu} \qquad (8)$$

### Comparison of Calculated and Measured Counting Rates for Cellophane

| | |
|---|---|
| Data | |
| Sample area, cm.$^2$ | 3.5 |
| Tritium activity in HTO, curies/g. | $1.54 \times 10^{-3}$ |
| Absorption coefficient for tritium beta particles in cellulose, $\mu$ (*7*), cm.$^{-1}$ | 4800 |
| Measured count rate, counts/sec. | 820 |

*Calculation of Counting Rate*

Cellophane was assumed flat, so from equation (8), and by assuming that

$$\frac{(T/H)_{OH \text{ in cellulose}}}{(T/H)_{water}} = 1.0,$$

$$\sigma_n = 590 \text{ particles/cm.}^2\text{/sec.}$$

The sample counting rate from equation (13) is:

$$N = 2100C$$

It is unlikely that $C$ is as low as 820/2100 for the counter studied; the calculated rate is probably too high because of the approximations in the assumptions of exponential absorption and in the estimate of $\mu$ (*7*).

### $\sigma_n$ for a Spherical Solid

Let point $O$ on the surface of sphere $C$ in Fig. 5 be the origin of coordinates. The method of calculating $\sigma_n$ is the same as above with the exception that the integration limit for $\theta$ is $\cos^{-1} r/2\rho$, where $\rho$ is the radius of the sphere. The flux $\sigma_n$ is therefore:

$$\sigma_n = \frac{K}{4\mu}\left(1 - \frac{1}{2\rho\mu}\right) \qquad (9)$$

To compare $\sigma_n$ for flat and spherical solids, $\mu$ was estimated to be 4800 cm.$^{-1}$ Therefore:

$$\sigma_n'' = \sigma_n'\left(1 - \frac{1}{\rho}\right) \qquad (10)$$

where $\sigma_n'$ and $\sigma_n''$ are the flux for flat solid and sphere, respectively. The results in Table I were calculated by substituting for values of $\rho$ in equation (10).

### Angular Distribution of Particle Velocities at a Flat Surface

The angular distribution of particle velocities at a surface is of importance in calculating the fraction of

Table IV. Calculated Angular Distribution of Velocities of Particles from a Flat Solid

| (Solid angle of cone)/$2\pi$ | Angle of cone to normal | Fraction of total radiation in cone |
|---|---|---|
| 0.13 | $\pi/6$ | 0.25 |
| 0.29 | $\pi/4$ | 0.50 |
| 0.50 | $\pi/3$ | 0.75 |
| 1.00 | $\pi/2$ | 1.00 |

particles emitted within a solid angle $\alpha$. In the following it is shown that the distribution is not random if rectilinear paths are assumed in the solid.

For rectilinear particle paths it was shown that equation (6) may be written as

$$\sigma_n = \frac{K}{4\mu} \sin^2 \theta_1$$

where $\theta_1 = \pi/2$ for a flat solid. The particles emitted at $O$ within a cone, with its axis normal to the surface and with an included angle $2\theta_1$, originate from the volume elements such that $\theta_1 > \theta > 0$. The flux contributed by the volume elements is $K/4\mu$ $(\sin^2\theta_1)$, and the solid angle included by the cone is $2\pi$ $(1 - \cos \theta_1)$.

Values of the fraction of particles included in cones of different solid angles are listed in Table IV and show that the greater density of particles is along the normal to the surface.

*Effect upon Counting Rates.* It is readily shown that the counting rate is independent of surface irregularities when rectilinear paths are assumed.

Let a point $O$ (Fig. 6) in the counter gas be considered as the origin of coordinates. The number of particles originating from the volume element $dV$ and having velocities directed to unit area (in the plane $\theta = \pm\pi/2$) at $O$ is:

$$dn = Ke^{-\mu(r-r_1)} \frac{\cos \theta}{4\pi r^2} dV \qquad (11)$$

where $r_1$ is the distance from $O$ to the solid surface in the direction of the volume element. The total particles $n$ emitted toward $O$ from the sample is given on integration of equation (11) by:

$$n = (K/8\pi\mu)(\phi_2 - \phi_1)(\sin^2\theta_2 - \sin^2 \theta_1) \qquad (12)$$

Equation (12) is independent of $r_1$ (determined by surface irregularities) and is determined only by sample size. Since the point $O$ is arbitrary, the total radiation emitted into the counter is independent of surface irregularities.

The assumption of rectilinear paths is undoubtedly invalid because particles are extensively scattered by solids. The measurements also showed that surface roughness did influence the counting rate as would be expected for a random distribution.

### *Calculation of Counting Rates for Models*

*Flat Solid.* For a flat solid $\alpha/2\pi = 1$ and $\sigma_{nB} = 0$. Therefore from equation (3)

$$N'' = C\sigma_n' A_S \qquad (13)$$

*Model I.* The counting rate was calculated for the projected area of model surface corresponding to the length $L$ (Fig. 2a) and unit width. The radiation is derived mainly from fiber $B$ and to a lesser extent from $C$, $D$, and $E$, which emit particles through the space between $A$ and $B$. Edge effects extend to distances of

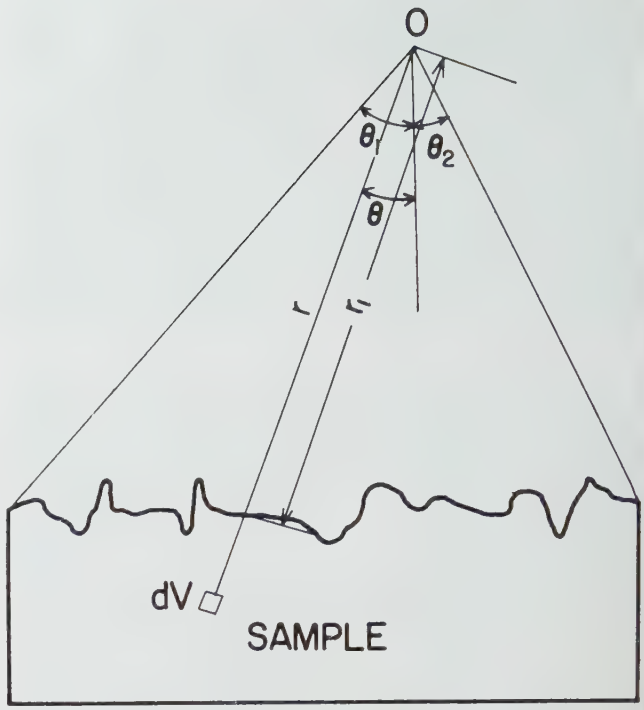


*Fig. 6. Coordinate system for calculating counting rates for rough solids*

(for details see text)

the order of $R(=5\mu)$ and can be neglected in the large samples (2.1 cm. in diameter) studied.

$N$ was estimated by numerical integration. The particles from fiber $B$ were calculated by summing average values for $\Delta A$ corresponding to unit length of the cylinder and an arc subtending 6° at the fiber axis. The shape of the solid angle $\alpha$ was that of a wide wedge which included angle $\gamma$ (Fig. 2a). The magnitude of $\gamma$ was readily determined from the geometry of the figure.

The number of particles contributed by $C$, $D$, and $E$ comprised a small fraction of the total and was estimated less accurately. A mean solid angle was taken as the average of the extreme values for each fiber and the counted particles estimated by equation (13).

The value of $(\sigma_n' + \sigma_{nB})$ was estimated by the following approximation. Of the flux $\sigma_n'$, 0.45 $\sigma_n'$ strikes adjacent cellulose surfaces (as calculated above). Back-scattering occurs with 10%, i.e., with $0.10 \times 0.45$ $\sigma_n' = 0.045$ $\sigma_n'$ particles. Therefore $(\sigma_n' + \sigma_{nB}) = 1.045$ $\sigma_n'$.

*Model II.* The fibers of thickness $Z$ (Fig. 2b) were spaced uniformly a distance $W$ apart to give a bulk density of 0.75 g. per cc. Back-scattering was neglected because its effect was found to be small in Model I and large variations in its magnitude would not be expected. Edge effects due to sharp corners of the fibers were approximately the same for values of $Z$ and $W$ in the range 5 to 25 $\mu$ (corresponding to $(Z/W)$ from 0.2 to 5.0), and were neglected.

The counting rate was calculated for the projected area corresponding to a length $2W$ and unit width. Particles emitted by the flat surface at the top of the fiber are given by $W\sigma_n'$. The particles from the cavity originate from the base and equally from each wall.

The base emits $W\sigma_n'$ particles of which a fraction is absorbed by the vertical walls. The fraction absorbed

by each wall for a given point on the base equals the fraction of the solid angle $2\pi$ obstructed by the wall. If $x$ is the vertical distance from a wall to a point on the base, the obstructed solid angle is 2 $\cot^{-1}$ $(x/Z)$ and has the shape of a wide wedge with included angle $\cot^{-1}$ $(x/Z)$. The number of particles from the differential area (1x $dx$) and absorbed by the wall is $(\sigma_n'/\pi)$ $\cot^{-1}$ $(x/Z)$ $dx$. The total number of particles from the base and absorbed by the two walls is:

$$n_{BA} = (2\sigma_n'/\pi) \int_0^W \cot^{-1}(x/Z)\, dx \qquad (14)$$

and the net particles emitted from the base and not absorbed is:

$$n_B = W\sigma_n' - (2\sigma_n'/\pi) \int_0^W \cot^{-1}(x/Z)\, dx \qquad (15)$$

The particles from the vertical walls are calculated as follows. Let $y$ be the vertical distance from the top of the fiber to any point on the wall. The corresponding solid angle unobstructed by cellulose is 2 $\cot^{-1}$ $(y/W)$ and has the shape of a wide wedge with the included angle $\cot^{-1}$ $(y/W)$. From equation (3) the counted paticles from the walls is:

$$N_W = C(2\sigma_n'/\pi) \int_0^z \cot^{-1}(y/W)\, dy \qquad (16)$$

Thus, $N$ for the area $2W$ is:

$$N = 2C\left[W\sigma_n' - (2\sigma_n'/\pi) \int_0^W \cot^{-1}(x/Z)dx + (2\sigma_n'/\pi) \int_0^z \cot^{-1}(y/W)dy\right] \qquad (17)$$

The data of Fig. 3 were calculated from equation (17).

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# Mechanisms of Slime Formation:

## *Fimbriae*

R. A. OPPERMANN and L. L. WOLFSON

**Bacteria are considered to be a major cause of paper mill "slime." The bacterial capsule has long been thought to be the primary cause of such deposits. However, there are protein fibrils that extend beyond the capsule. These fimbriae are reported to have adhesive properties for cellulose and other materials. One hundred deposits from 53 paper mills have been examined for fimbriated bacteria. One or more fimbriated species were found in 83% of the samples. Many of the bacterial genera found in mill "slimes" possessed fimbriae when grown as pure cultures. Disks of stainless steel and lens tissue were placed in suspensions of radioactive, positively or negatively fimbriated cells. The amount of cells adhering to cellulose was constant after the first hour while on stainless steel, the numbers reached a constant value after 6 to 8 hr.**

THE CLOSING of paper mill water systems for economic or antipollution purposes engendered a new problem, that of "slime" deposits. These deposits are classed as (1) microbiological or (2) inorganic. Examination of biological deposits shows large numbers of bacteria, fungi, and/or yeasts with fines and crystalline material. Chemical or inorganic deposits are usually found to contain pitch, alumina, clays, hydrated silica, resins, iron sulfides, and fine fibers. However, most paper mill "slimes" are combinations of microbiological and chemical materials. The proportion of the components in a deposit may vary from mill to mill and so may the appearance and consistency.

The required "nucleus" for the agglomeration of deposits is considered to be the bacterial cell. Therefore, research would be focused primarily on the cell surface for the causes of "slime" deposits. Several investigators have selected the bacterial capsule or slime layer as this cause (*1, 2*). However, direct examination of many, but not all, white waters and deposits shows few encapsulated bacteria.

While the capsule has long been thought to be the adhesive that binds "slime," there are other surface features that might account for deposit formation. Extremely fine protein fibrils have been recently reported that extend beyond the capsule and, therefore, would be in a better position for attachment to fibers and other organisms. These fimbriae are thought to be organs of attachment (*3*) and have been reported to be adsorbed onto and to agglomerate cellulose fibers and other materials (*4*). It is possible that deposits showing many bacteria, but few encapsulated forms, could be caused and cemented together by means of fimbriated bacteria. To examine the hypothesis that fimbriae are a cause of paper mill "slimes" the following experiments were designed and performed.

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### MATERIALS AND METHODS

#### *Examination of Slimes*

Paper mill slimes were obtained from many mills and were examined for the presence of fimbriated bacteria by one of the three different methods which follow:

(1) Total counts were made from the original deposit material using Difco Tryptone Glucose Extract

(TGE) Agar. From three to five colonies of the most prevalent types found were inoculated into nutrient broth containing 0.85% NaCl. The bacteria were incubated at 37°C. for 24 hr. and centrifuged out of the medium. The cells were resuspended in sterile deionized (D.I.) water and recentrifuged. The cells were again suspended in sterile D.I. water and placed on electron microscope specimen grids which had been coated with a Parlodion (Mallinckrodt Chemical Works, St. Louis, Mo.) film. Exposure of the grids to the vapors of osmic acid (osmium tetroxide) for 5 min. was used to fix and "stain" the cells. The prepared grids were lightly shadowed with chromium at an angle of approximately 20° and examined at a magnification of 17,500× in an R.C.A. Model EMU-2-D, 50 K/V electron microscope.

(2) Colonies of *Aerobacter* and *Flavobacterium* were selected from total count plates and inoculated into saline nutrient broth, etc., as above.

(3) Specimen grids filmed with Parlodion and attached to a glass slide were immersed in a suspension made from the raw deposit by shaking 1 g. of the deposit in 99 ml. of sterile D.I. water. The course particles were allowed to settle and the slide placed vertically into the suspension for 2 hr. Fimbriated cells would attach themselves to the cellulosic films which were dried, shadowed, and examined as above.

A deposit was termed positive for the presence of fimbriae ($F^+$) if any of the cultures examined were fimbriated. When using Method 3, approximately 10% or more of the cells examined had to possess fimbriae to be designated as positive. The same was true of any single culture produced by the other two methods listed.

### *Radioactive Methods*

Two species of bacteria were used, *Aerobacter aerogenes* ATCC no. 8308, and *Flavobacterium sp* isolated from a mill deposit. The *Aerobacter* culture was highly fimbriated over the entire surface of the cell. The *Flavobacterium* possessed few fimbriae, these mainly at one end of the cell. Both cultures contained nonfimbriated varieties. Nonfimbriated *Flavobacterium* were obtained using the method of Duguid and Gillies *(5)*. *Aerobacter* resisted attempts to isolate nonfimbriated types. Flagella or capsules were not found to be present by electron microscope examination of the cultures.

Five cultures were used to test fimbriate adhesion: (A) a $F^+$ *A. aerogenes*, (B) a killed $F^+$ *A. aerogenes*, (C) a $F^-$ (nonfimbriate) *Flavobacterium*, (D) a $F^+$ *Flavobacterium*, and (E) a killed $F^+$ *Flavobacterium*. The killed cells were produced by adding formalin to 0.4% to a normal $F^+$ culture.

*Growth of Radioactive Cells.* The five cultures were grown in a medium composed of:

| | |
|---|---|
| Glucose | 0.25 g. |
| Asparagine | 0.10 g. |
| Sodium chloride | 0.20 g. |
| Monopotassium phosphate | 0.05 g. |
| Magnesium sulfate | 0.025 g. |
| Deionized water Q.S. to 100 ml. | |

The pH of the medium was adjusted to 7.0 with sodium hydroxide before autoclaving at 15 p.s.i. for 15 min. Just prior to sterilization, 10 microcuries of uniformly labeled glucose $C^{14}$ were added to each of 5 flasks, each containing 25 ml. of medium.

The *A. aerogenes* cultures were incubated at 37°C. for 24 hr., while the *Flavobacterium* cultures were incubated at 30°C. After incubation, the cells were centrifuged out of the growth medium and resuspended in 15 ml. of Krebs-Ringer phosphate (KRP) solution pH 6.5. After a second washing, 1 ml. of the cultures (B) and (E) were placed in 99 ml. sterile dilution blanks for a total count of the cells present. The suspensions (B and E) were then made up to 0.4% with 40% formalin. The live cells were centrifuged and washed twice more. The killed cells were centrifuged and resuspended twice with 0.4% formalin in KRP solution and finally washed twice with KRP solution to remove the formalin. All suspensions were made up to 15 ml. with KRP. One milliliter each of cultures A,C, and D was removed for total counts.

Total counts were made of each culture using TGE agar as the growth medium. Simultaneously decimal dilutions were placed on counting planchets for radioactive analysis. A disk of lens tissue (Schaar & Co., Chicago, Ill.) was placed in the planchet to prevent the aliquots from becoming concentrated in one spot. The agar plates were incubated at 37°C. for *Aerobacter*, and at 30°C. for *Flavobacterium*, for 48 hr. The standard planchets were allowed to dry at room temperature, and a paper spacer was placed over the lens tissue disk before stacking, to prevent buckling of the disks.

*The Adsorption Test.* Each test set contained nine planchets containing 4 ml. of KRP solution and one lens tissue disk 2 in. in diameter. One milliliter of radioactive bacterial suspension was added to each planchet, which was then covered with the top of a Petri dish. This procedure was repeated with each culture. Adsorption was allowed to take place for

**Table I**

| *Culture* | *Cells/ml.* | *Count/min./ml.* |
|---|---|---|
| A | $3.4 \times 10^8$ | 23,000 |
| B | $2.97 \times 10^8$ | 16,400 |
| C | $6.1 \times 10^7$ | 9,000 |
| D | $2.27 \times 10^8$ | 11,600 |
| E | $2.85 \times 10^8$ | 10,400 |

1, 2, 3, 4, 6, 8, 10, 12, and 24 hr. At the end of any one time period, the cell suspension was poured out of the planchet, and the paper disk was held to the bottom while it was washed twice with 20 ml. of D.I. water. The lens tissue was floated off the planchet by immersing it in D.I. water. A second planchet was used to receive the paper disk. Both planchets (with and without tissue disk) were allowed to dry at room temperature.

Removal of the tissue disk allowed a determination to be made of the adsorption of the cells to the metal surface of the "reaction planchet" as well as to cellulose. The planchets were stainless steel, 2 in. in diameter with a rim (Nuclear Measurements Corp., Indianapolis, Ind.). All determinations of radioactivity were made using a proportional counter converter Model PCC-10A (Nuclear Measurements Corp., Indianapolis, Ind.). Used at a voltage of 1800 v. the efficiency was 50%.

The total counts made in the agar plates are given in Table I, with the amount of radioactivity of the suspension.

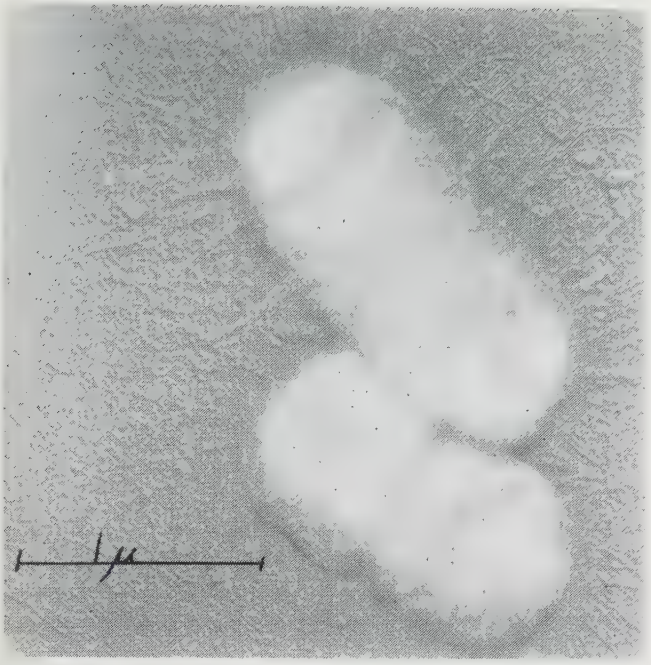

*Fig. 1. Electronmicrograph of the surface of* Aerobacter aerogenes. *The hazy area was caused by the presence of a capsule*

From the data, graphs were made to allow a determination of the number of cells on a disk when the radioactivity of the specimen was known. It had been found earlier that no increase or great decrease in the cells took place in KRP solution in 24 hr.

## RESULTS AND DISCUSSION

Observation by electron microscope showed two types of fimbriation occurring in paper mill deposits. The first, typified by *A. aerogenes*, (Fig. 1) showed that fimbriae existed all over the entire surface, numbering from 50 to several hundred per cell (*4*). In the second type, shown usually in long rods such as *Flavobacterium*,

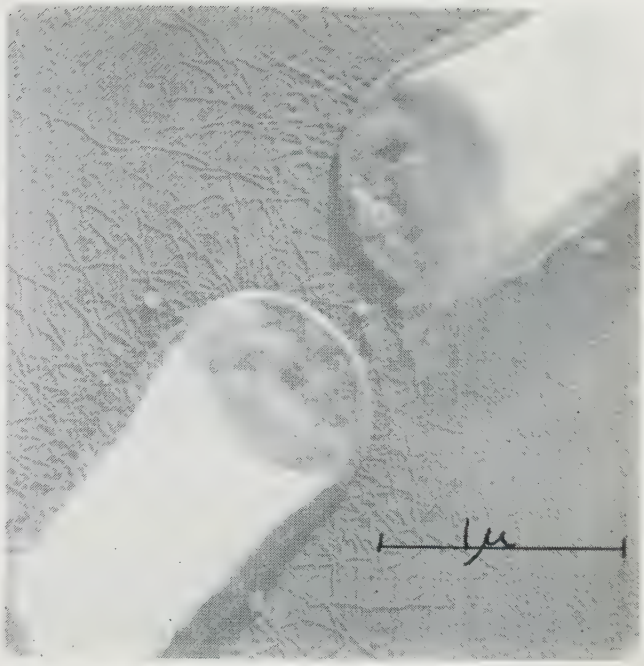

*Fig. 3. Electronmicrograph showing the tangle of fimbriae from two cells in close proximity to each other*

the fimbriae are few in number and located at one or both ends of the cell (Fig. 2).

The slight opacity immediately around the cell in the micrograph of *A. aerogenes* was caused by the presence of a capsule. The fimbriae can be seen to pass through the capsule and extend some distance beyond. Fimbriae were distinguished from flagella by their shorter length, small width (approximately 0.01 $\mu$), and the absence of a flagellar type wave form. Examination of the micrographs indicated that fimbriae are real appendages of the cell and not "strings" formed by the extrusion of the cell contents or slime layer in drying. If they were formed during the drying of the liquid medium, they would point primarily in the direction of

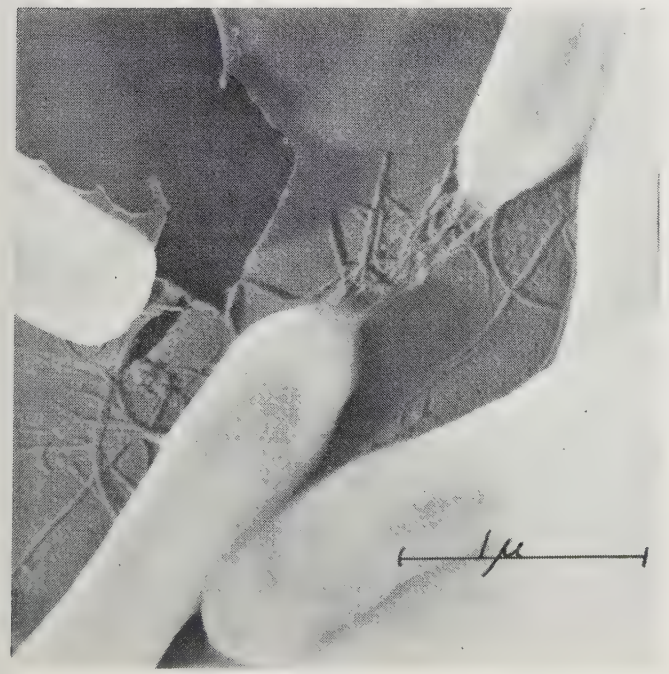

*Fig. 2. Electronmicrograph of a group of* Flavobacterium *species cells*

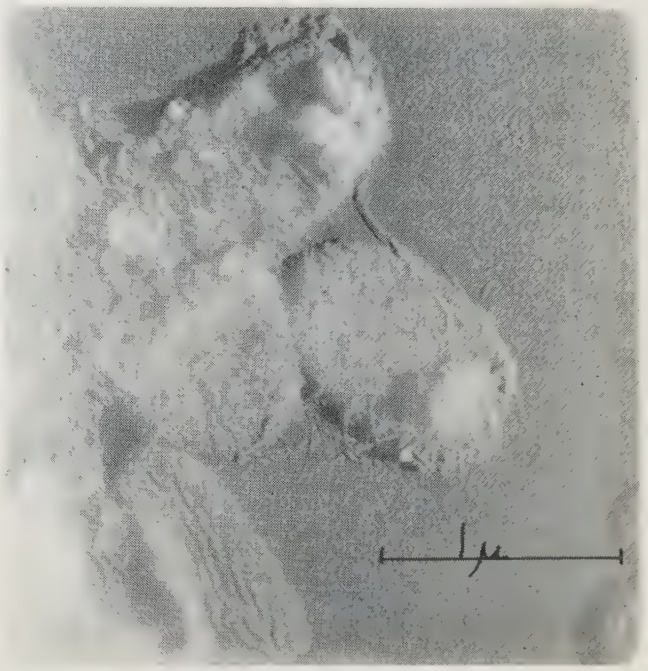

*Fig. 4. Electronmicrograph of a section of a "mill deposit" formed in the laboratory*

the liquid flow caused by uneven drying, and would flow into and join each other. But, as shown, fimbriae extend in all directions and cross over and under other fimbriae.

The use of fimbriae to form clumps of bacteria is shown in Figs. 2 and 3. This junction may be caused either through a simple matting or a chemical and/or physical attraction of the fibrils. Figure 4 shows a deposit formed in a laboratory white water using *A. aerogenes* as the principal organism. The bacteria can be seen to be joined by short thick fimbriae.

In all, 100 slimes from 51 paper mills have been examined. Of this number, 83% contained from 1 to 3 fimbriated species, 10 contained none, and 7 contained bacteria with doubtful fimbriation. The extreme fineness of some fimbriae combined with a granular background shadowing occasionally made positive identification of fimbriae impossible.

Three methods were used to show the presence of fimbriated bacteria in deposits: (1) random picking of colonies, (2) selection of *Aerobacter* and *Flavobacterium* species, and (3) adsorption of cells to a cellulosic film. An analysis of the methods appears in Table II. Most of the negative "slimes" were found using Method I. When it was discovered that *Aerobacter* and *Flavobacterium*, two of the bacterial genera most often found in large numbers in paper mill systems, were fimbriated, it became more convenient to examine only these organisms when found. This procedure produced fewer negative deposits than the random sampling of the many species that might be found in a slime.

**Table II. Distribution of Results of Deposit Examination for Fimbriae**

| Method | Deposits examined | Positive | Negative | Doubtful |
|---|---|---|---|---|
| (1) Random | 37 | 24 | 9 | 4 |
| (2) Selective | 36 | 33 | 1 | 2 |
| (3) Adsorptive | 27 | 26 | 0 | 1 |
| Totals | 100 | 83 | 10 | 7 |

Procedure 3 was devised to show (1) that fimbriated bacteria were present in "slimes," and (2) as a "quick test" for the detection of fimbriated organisms in a deposit. Theoretically, if fimbriae are important for bacterial adhesion to cellulose, those cells with fimbriae would attach themselves to a cellulosic surface faster and in greater quantity than those cells without such a mechanism. The film was placed vertically in the suspension to decrease the number of cells that might settle and form a loose attachment due to electrostatic charge or capsules. The film was washed with D.I. water to remove those organisms not firmly attached, but only adhering loosely. From the table it can be seen that the number of negatives and doubtful deposits was greatly decreased. Approximately 80% of the cells found on the film were fimbriated, although the total number was not large.

**Table III. Bacterial Genera from Paper Mill Deposits Containing Fimbriated Species**

| | |
|---|---|
| *Aerobacter* | *Flavobacterium* |
| *Chromobacterium* | *Klebsiella* |
| *Clostridium* | *Proteus* |
| *Escherichia* | *Pseudomonas* |
| *Serratia* | |

Examination of cultures of bacteria derived from paper deposits showed fimbriated species in the genera shown in Table III. From the list it would seem that most all of the genera usually present in a mill deposit contain fimbriated species. It is interesting to note that most of the organisms found in "slime" were gram negative and a great many were found to be fimbriated.

The above data were all of an indicative nature, but proof that fimbriae are a cause of paper mill slime was lacking. Accordingly, an attempt was made to determine the numbers and rate of attachment of fimbriated bacteria to cellulose and stainless steel.

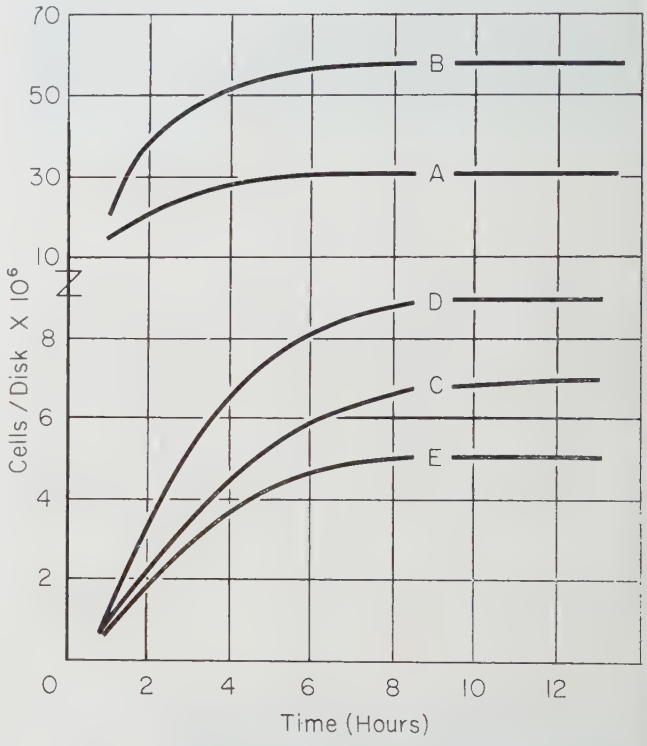


***Fig. 5. Adsorption of fimbriated* Aerobacter aerogenes *and* Flavobacterium *species to a stainless steel substratum. The five cultures are: (A) $F^+$* A. aerogenes, *(B) formalin killed $F^+$* A. aerogenes, *(C) $F^-$* Flavobacterium, *and (D) formalin killed $F^+$* Flavobacterium *(E) $F^+$* Flavobacterium

Lens tissue was used as a cellulose substrate to give a large surface for adsorption. The planchets used were stainless steel, and therefore could be used as the metal substrate. The test was performed in the planchet and so allowed two samples to be prepared at one time.

The data presented in Table IV show the results of the test for fimbriated bacterial adsorption to cellulose. Since the greatest adsorption of cells occurred within the first hour of contact, and longer experimental periods did not change the results greatly, they are listed as the average number adsorbed in the cumulative time period. The death of the cell by formalin did not decrease its adhesion to cellulose. Adsorption did not seem to depend on a reaction of living protein or protoplasm. Clear evidence for fimbriae as cause of "slime" did not appear because the nonfimbriated *Flavobacterium* adsorbed as strongly as the fimbriated form.

A nonfimbriated *Aerobacter* was not used due to the great difficulty in maintaining a nonfimbriate substrain.

The stainless steel disks offered differential effects. Figure 5 shows that the amount of *A. aerogenes* $F^+$ cells adsorbed was relatively constant after the first 4 hr. The number of $F^+$ *Flavobacterium* became constant after 8 hr., while that of the $F^-$ cells was slowly increasing during the total time period. The adsorption of *Flavobacterium* $F^+$ or $F^-$ did not reach the amount of *Aerobacter* found during the first hour. The

**Table IV. Numbers of Bacteria Adsorbed on Lens Tissue**

| | *Cells/2 in. disk* |
|---|---|
| *A. aerogenes* | |
| $F^+$ | $3.9 \times 10^6$ |
| $F^+$ killed | 3.5 |
| *Flavobacterium* | |
| $F^-$ | 4.3 |
| $F^+$ | 4.0 |
| $F^+$ killed | 5.0 |

final number of *Aerobacter* adsorbed to metal was approximately 10 times that attached to cellulose. The average amount of *Flavobacterium* adhesion was almost the same for the two materials, the major difference being that adsorption to metal was gradual at first, and the final number greater than those found on cellulose.

There was a noticeable difference in adsorption between live and killed bacteria. Through the use of the electron microscope it was noted that formalin seemed to have a slight physical effect on fimbriae of *Flavobacterium* but not *Aerobacter*. The outlines of the strands were not as sharp, nor were they as thin as untreated fimbriae. This appearance may have been due to a chemical change which could have caused the difference found for metal adhesion between killed and live cells. Something similar may have been active in cellulose adhesion, but the data are not clear enough to indicate this.

Electron microscope examination of the $F^-$ *Flavobacterium* culture showed the presence of a moderate number of fimbriated forms. There seemed to be a partial reversion to the original culture of mixed types, which could account for some of the equality of adsorption to cellulose of the $F^+$ and $F^-$ cultures. No reason can be given at this time for the similarity of the average results for the adsorption of the five cultures to cellulose. Some other method of adhesion, not making use of fimbriae, may also have been operative, such as electrostatic attraction, or the presence of undetected capsules. The like adsorption to cellulose could suggest a limited number of sites for adhesion of the protein fibrils.

At a pH of 6.5, it was apparent that the attachment of the fimbriae was greater to stainless steel than to cellulose. If the mechanism of attachment makes use of hydrogen bonding between carboxyl, hydrogen, and amine groups on the fimbriae and hydroxyl, hydrogen, and oxygen bridges in cellulose, a change in pH could alter this pattern of effects. Protein configuration is known to vary with changes in pH, so it is possible that at a higher pH (4.0–5.0) a modification in the adsorption to cellulose might take place. Various concentrations of salts or colloidal material in the medium could likewise affect the results. Such a shift of protein configuration directed by local conditions, and the concomitant differences in adhesion to materials, could explain the texture differences and randomness of mill deposits in a single mill or system. Each new addition could change the properties of the fimbriae or cellulose, resulting in different deposits on the same material substrate.

Lack of primary attraction to cellulose fibers is an advantage to mill bacteria. They would otherwise be attracted to the cellulose fibers and swept out of the mill in the paper sheet faster than they multiply. But with a greater adhesion to metal, glass, etc., they could form a film on that surface, capturing from the passing suspension bits of fiber and other cells to eventually form a deposit. The adsorption of organic matter from the water onto the surface of the material would supply nutrient to the growing deposit (*6*). The large amounts of clay and/or titanium dioxide found in biological "slimes" could aid in gathering food by selective adsorption, or may be preferential substrates on which to begin "slime" formation.

Although the above experiments did not conclusively prove the initial hypothesis, there is strong indicative evidence as well as theory to indicate that fimbriae are involved in the formation of paper mill "slime." A refinement of methods, as well as new tests for adsorption to clays, etc., under a variety of conditions of pH and additive concentration should yield a complete theory with adequate proof.

## SUMMARY

The examination of 100 paper mill "slimes" from 51 mills showed that 83% contained fimbriated bacteria. In 11% of the samples no fimbriated forms were found and 6% contained organisms of doubtful fimbriation. When grown as pure cultures, most of the bacterial genera usually present in a mill deposit showed fimbriated species.

Suspensions of fimbriated *A. aerogenes*, and positively and negatively fimbriated *Flavobacterium*, were adsorbed in approximately equal numbers to cellulose. There was a tenfold increase for *A. aerogenes* attachment to stainless steel. The amounts of *Flavobacterium* adsorbed to metal began at low levels, but ended in a greater number than were attached to cellulose. The number of cells adhering to cellulose was constant after the first hour, while on stainless steel the amounts increased with time and reached a constant number after 6 to 8 hr. Hydrogen bonding is postulated as the mechanism of attachment. The implications of fimbriae and different types of "slimes" are briefly discussed.

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# Evaluation of Seed Galactomannans from Legumes as Paper Sizes

H. L. TOOKEY, A. J. ERNST, R. L. LOHMAR, AND I. A. WOLFF

**Seed galactomannans from *Crotalaria intermedia*, *C. spectabilis*, *Cassia emarginata*, *Trigonella foenum-graecum*, and *Melilotus indica* were tested as wet-end additives for softwood kraft and sulfite pulps. The first three were generally equivalent to commercial guar gum in improving burst, tensile, and fold properties of handsheets. The *Trigonella* and *Melilotus* mucilages improved the strength characteristics of handsheets, but were not as effective as guar gum. All the galactomannans tested as surface sizes were as effective as guar or locust bean gums in improving strength characteristics of a commercial coating base.**

ONE OBJECTIVE of research in the U. S. Department of Agriculture is to find new industrial raw materials among the many plants that have had little or no study of their chemical constituents (*1*). Included in this program is the investigation of water-soluble carbohydrate mucilages. A survey of seeds of 175 plant species has recently been reported (*2*). Seed from several legume species contain sufficient mucilage to warrant their further evaluation. In this paper we report a preliminary study of galactomannan mucilages from five species. Although several have been mentioned as sources of seed mucilage (*3*, *4*), the mucilages have apparently not been tested as paper additives (*5*, *6*). Since guar gum, also a galactomannan obtained from the seed of an annual legume, has had increasing acceptance in the paper industry within the past decade, it was adopted as a standard for comparison. Some comparisons were also made with locust bean gum.

## MATERIALS AND METHODS

### *Mucilages*

Mucilages were isolated from ground seeds by extraction with hot water (*2*). The mucilage solution, approximately 0.5%, was chilled and made 0.5 *M* in trichloroacetic acid. This mixture was allowed to stand in the refrigerator 2 hr., then centrifuged to remove coagulated protein. The mucilage was precipitated from the supernatant solution by adding four volumes of ethanol, separated, washed with 95% ethanol, and dried (Table I).

Guar and locust bean gums were technical grade commercial products. One mucilage sample from *Crotalaria intermedia* was endosperm flour separated from the seed by dry milling.

One-per-cent solutions were made by dispersing air-dry mucilage in tap water at 50 to 55°C. and agitating for 10 min. The solutions were allowed to stand for 1 to 2 hr. before they were mixed with the pulp. Solutions of 0.5 or 1.0% were used for surface sizing.

### *Viscosity*

Viscosities of the mucilage solutions were measured with a Brookfield viscometer, model LVF, at 30 r.p.m. and 25°C.

### *Pulps*

Softwood unbleached kraft pulp was refined in a Valley beater to a Schopper-Riegler freeness of 860 ml., to develop approximately 55% of its maximum bursting strength. Softwood bleached sulfite pulp was beaten to a freeness of 760 ml., to develop approximately 70% of its maximum bursting strength.

### *Wet-End Addition*

Mucilage solutions were added to 0.3% pulp suspensions in tap water and agitated 5 min. before preparation of handsheets. The level of additive was based on dry mucilage and moisture-free pulp. When rosin (1%) and alum (2%) were used, the pulp suspension was adjusted with sulfuric acid to pH 5.0 to 5.2 before the addition of mucilage. Handsheets of 1.2 g. were prepared and tested according to TAPPI standards. Retention of additives in the handsheets was not measured.

### *Surface Sizing*

Mechanically operated rubber rolls with attached size bath maintained between 50 and 55°C. were used to spread a uniform coat of mucilage on a commercial magazine coating base. The sized sheets were drum-dried at 100 and 105°C., conditioned, and tested.

**Table I. Mucilages Evaluated as Paper Sizes**

| *Mucilage source* | *Common name* | *Nitrogen, % dry basis* | *Mucilage isolated from seed, %* | *Viscosity of 1% soln., cp.* | *Mannose: galactose ratio* |
|---|---|---|---|---|---|
| *Cassia emarginata* | Senna | 1.05 | 29 | ... | 2.7 |
| *Crotalaria intermedia* | Rattlebox | 1.99 | 26 | 84 | 2.8 |
| *Crotalaria spectabilis* | Rattlebox, showy crotalaria | 2.51 | 24 | .. | 2.8 |
| *Melilotus indica* | Annual yellow sweet clover | 0 80 | 23 | ... | 1.0 |
| *Trigonella foenum-graecum* | Fenugreek | 0.99 | 24 | .. | 1.2 |
| *Cyamopsis tetragonolobus* (commercial) | Guar | 1.30 | ... | 1650 | 1.8 |
| *Ceratonia siliqua* (commercial) | Locust bean | ... | ... | 1360 | 3 to 6 |
| *Crotalaria intermedia* (endosperm flour) | Rattlebox | 0.65 | 17 | 2080 | 2.8 |

H. L. TOOKEY, A. J. ERNST, R. L. LOHMAR (deceased), and I. A. WOLFF, Northern Regional Research Laboratory, Peoria, Ill. This is a laboratory of the Northern Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture.

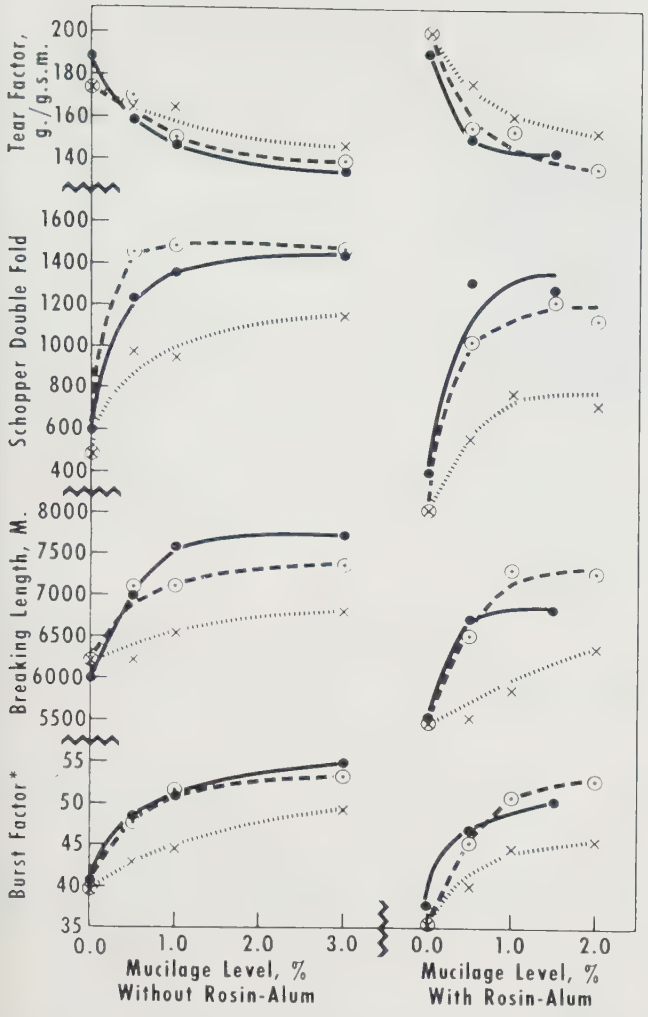


*Fig. 1. Strength characteristics of softwood unbleached kraft pulp with* Crotalaria *mucilages as wet-end additives. * Burst factor in g./cm.²/g.s.m.*

●———●, *Crotalaria intermedia.* ⊙— — —⊙, *Crotalaria spectabilis.* x- - - -x, Guar (commercial).

## RESULTS AND DISCUSSION

### *Wet-End Addition to Kraft Pulp*

Increases in burst and tensile strength and in fold properties of handsheets were marked when mucilages from either of the *Crotalaria* species were added to kraft pulp (Fig. 1). Strength data at the 5.0% level of addition were essentially the same as those at the 2.0 and 3.0% level, so are not reported. The improvements were superior to those obtained with commercial guar gum. As expected, with increased burst strength there was some decrease in tear factor. Similar improvements were noted when the mucilages were used in combination with rosin and alum (Fig. 1); mucilages at the 0.5% level restored the strength to values well above that of the pulp alone with no rosin or alum. Little additional benefit was achieved at mucilage levels above 1%, with or without rosin and alum. There was not much difference between the performance of mucilages from the two *Crotalaria* species; this result reflects their essential structural similarity. Fiber from the stalks of some *Crotalaria* species have been examined as raw material sources of pulp for papermaking (*7–9*). The favorable results of those studies indicate that selected *Crotalaria* species may serve as dual-purpose crops for use in the paper industry.

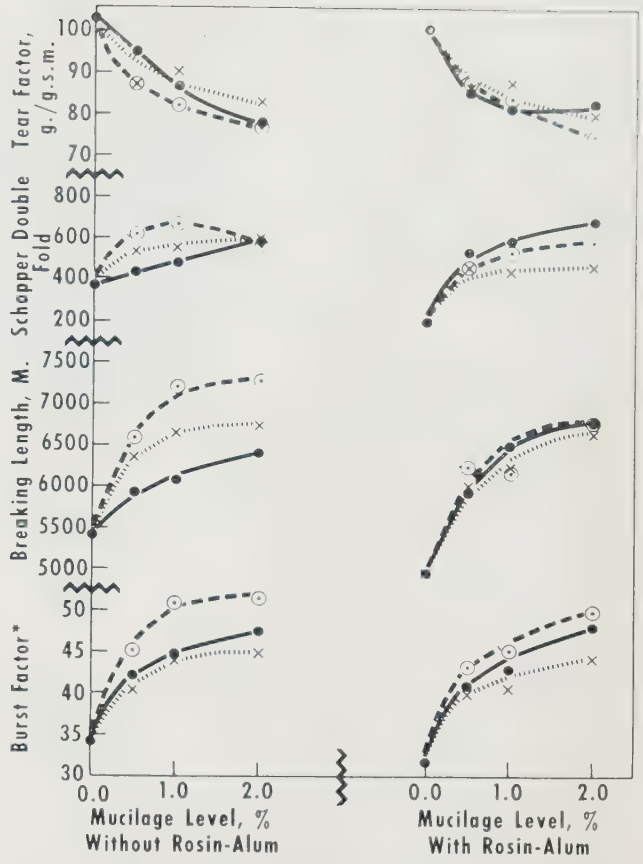


*Fig. 2. Strength characteristics of softwood bleached sulfite pulp with* Crotalaria *mucilages as wet-end additives. * Burst factor in g./cm.²/g.s.m.*

●———●, *Crotalaria intermedia.* ⊙— — —⊙, *Crotalaria spectabilis.* x- - - -x, Guar (commercial).

### *Wet-End Addition to Sulfite Pulp*

Results obtained when softwood bleached sulfite pulp was sized with the *Crotalaria* mucilages were less consistent (Fig. 2). *C. spectabilis* mucilage was more effective than guar gum, but the mucilage from *C. intermedia* was generally comparable to that from guar, except in breaking length for which it gave somewhat less improvement than guar. When these mucilages were used in combination with rosin and alum, there was less divergence in properties of the sized handsheets.

Comparisons were also made with three other mucilages, *Cassia emarginata*, *Melilotus indica*, and *Trigonella foenum-graecum*. The *Cassia emarginata* mucilage gave the greatest improvement over guar in burst, tensile, and fold among the three mucilages (Fig. 3). The *Melilotus indica* mucilage gave the least improvement in strength and was inferior to guar, although it gave the best improvement in fold and the least decrease in tear factor. Only minor changes were noted in water absorbancy as the levels of any of the various mucilages were increased.

### *Endosperm Flour of* C. intermedia

Because of the promising performance of the *Crotalaria* mucilages, limited comparisons were made between commercial guar gum and endosperm flour isolated from *C. intermedia* seeds by a dry-milling process. This mucilage improved the strength and fold characteristics of softwood sulfite pulp slightly more than guar gum (Fig. 4), accompanied, as usual, by a decrease in tearing resistance.

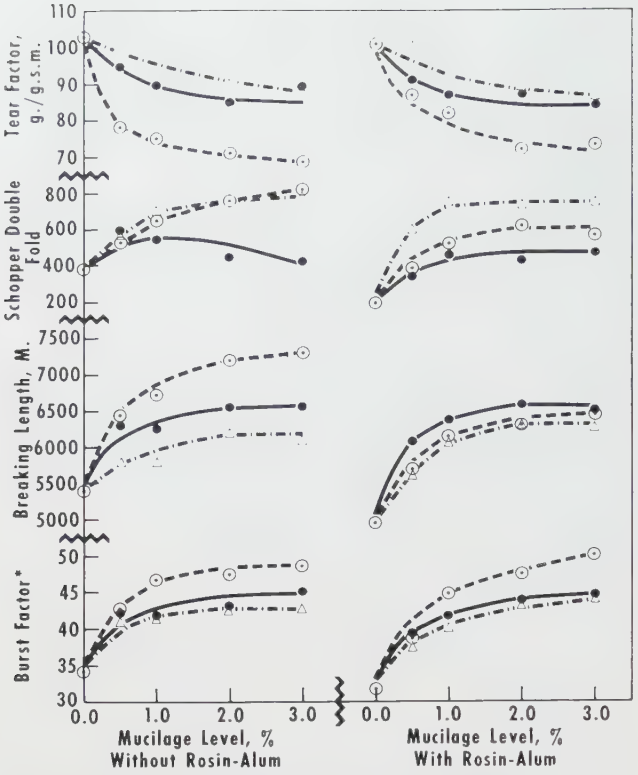


*Fig. 3. Strength characteristics of softwood bleached sulfite pulp with other galactomannan mucilages as wet-end additives. * Burst factor in g./cm.²/g.s.m.*

●———●, *Trigonella foenum-graecum.* ⊙— — —⊙, *Cassia emarginata.* △·-·-·-·-△, *Melilotus indica.*

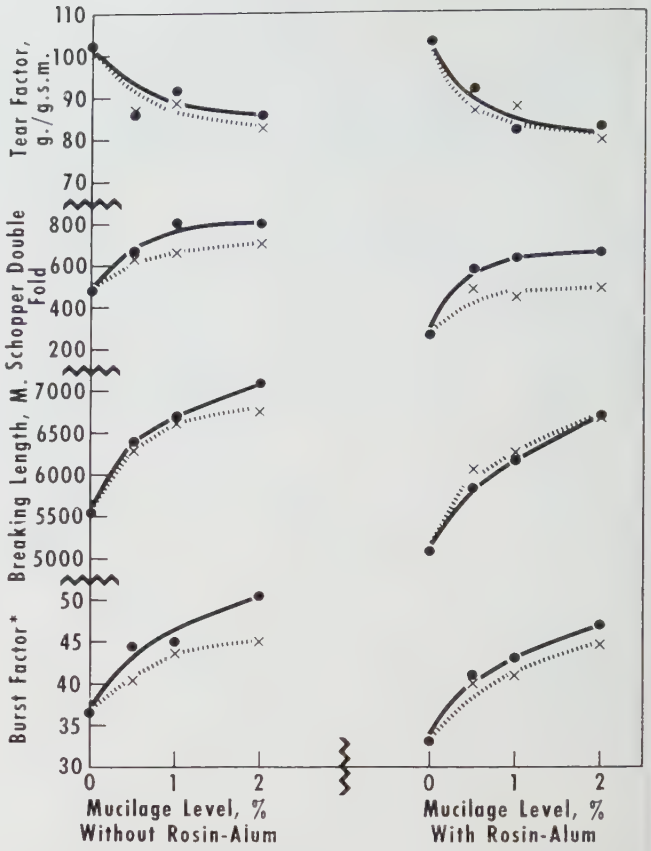


*Fig. 4. Strength characteristics of softwood bleached sulfite pulp with endosperm flour of* **Crotalaria intermedia** *seed as wet-end additive. * Burst factor in g./cm.² g.s.m.*

●———●, *Crotalaria intermedia.* x- - -x, Guar (commercial).

The performance of the endosperm flour is similar to that of mucilage extracted with water from *C. intermedia* seed (Fig. 2), yet the viscosities of the two solutions are quite different (Table I). The lowered viscosity of the water-extracted mucilage is caused by the action of enzymes present in the seed.

### *Surface Sizing*

Four of the galactomannan mucilages were compared with commercial guar and locust bean gums as surface sizes (Table II). The sheets retained approximately 2% mucilage by weight. No chemical analysis of retention was made. There was no consistent ranking of the galactomannans in the various tests, but the two *Crotalaria* mucilages and those from *Trigonella foenum-graecum* and *Cassia emarginata* were at least as effective as the commercial products in improving strength characteristics.

Apparently a general relationship exists between mannose to galactose ratio and wet-end sizing efficacy of the galactomannans. Those with ratios approaching 3, *Cassia emarginata* and the *Crotalarias,* appear to impart a higher bursting strength than guar, which has a ratio ≲ 2. *Melilotus indica* and *Trigonella foenum-graecum,* with ratios near 1, are slightly inferior to guar. However, there may be more subtle differences as yet uninvestigated among the galactomannans. A similar correlation for surface-sizing properties is not evident.

**Table II. Strength Characteristics of Paper Surface-Sized with Galactomannan Mucilages**

| *Mucilage* | *Size solution conc., %* | *Burst factor, g./cm.²/ g.s.m.* | *Tear factor, CD, g./g.s.m.* | *Breaking length, MD, m* | *Fold, Schopper* |
|---|---|---|---|---|---|
| No mucilage | ... | 15.7 | 89 | 4340 | 42 |
| *Cassia emarginata* | 1.0 | 24.3 | 79 | 5550 | 86 |
| *Crotalaria intermedia*[a] | 0.5 | 22.5 | 82 | 5120 | 66 |
| *Crotalaria intermedia*[a] | 1.0 | 23.6 | 81 | 5360 | 100 |
| *Crotalaria spectabilis* | 1.0 | 24.3 | 81 | 5360 | 64 |
| *Trigonella foenum-graecum* | 1.0 | 22.0 | 77 | 5450 | 69 |
| Guar (commercial) | 0.5 | 21.1 | 90 | 4830 | 55 |
| Guar (commercial) | 1.0 | 23.7 | 78 | 5360 | 67 |
| Locust bean (commercial) | 1.0 | 22.0 | 79 | 5310 | 69 |

[a] Water extracted.

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# ASSOCIATION NEWS AND EVENTS

## *National Conferences*

*1962*

*Forty-seventh Annual Meeting*, Feb. 18–22, 1962, Commodore Hotel, New York, N. Y.

*Thirteenth Coating Conference*, May 14–16, 1962, Netherland-Hilton Hotel, Cincinnati, Ohio.

*Thirteenth Testing Conference*, Sept. 18–20, 1962, Benjamin Franklin Hotel, Philadelphia, Pa.

*Seventeenth Plastics-Paper Conference*, Oct. 8–11, 1962, Statler-Hilton Hotel, Boston, Mass.

*Seventeenth Engineering Conference*, Oct. 14–19, 1962, Queen Elizabeth Hotel, Montreal, Que.

*Twelfth Corrugated Containers Conference*, Oct. 23 26, 1962, Statler-Hilton Hotel, Boston, Mass.

*Sixteenth Alkaline Pulping Conference*, Oct. 31, Nov. 1–2, 1962, DeSoto Hotel, Savannah, Ga.

*1963*

*Fourteenth Coating Conference*, May 19–22, 1963, Concord Hotel, Kiamesha Lake, N. Y.

*Thirteenth Corrugated Containers Conference*, Oct. 22–25, 1963, Statler-Hilton Hotel, Detroit, Mich.

*Eighteenth Engineering Conference*, Oct. 28–31, 1963, Netherland-Hilton Hotel, Cincinnati, Ohio.

## *1962 Annual Meeting, Technical Section Canadian Pulp and Paper Association*

The 1962 Annual Meeting of the Technical Section, Canadian Pulp and Paper Association will take place at the Queen Elizabeth Hotel, Montreal, on the last Tuesday, Wednesday, Thursday, and Friday, of January, the 23, 24, 25, and 26. Sessions will begin at 2:00 p.m. on Tuesday and will end at noon on Friday; the Registration Desk will open at 9:00 a.m. on Tuesday. A products display will be held concurrently with the meeting.

Requests for accommodation should be addressed to Nelson Verge, Rooms Division Manager of the hotel. Give the date and time of your arrival, and tell the hotel if you are a member of the Technical Section. The Technical Section does *not* handle room reservations.

Anyone wishing to present a paper at this meeting should contact Douglas Jones, Executive Secretary, Technical Section, Canadian Pulp and Paper Association, 2280 Sun Life Building, Montreal 2, Que., at an early date.

## *Southern California District of TAPPI Organized*

Members of TAPPI in Los Angeles have organized the Southern California District of the Golden Gate Section, according to an announcement by H. M. Annis, president of the Association. The petition for district status was approved by the TAPPI Executive Committee and the Local Section Operations Committee at their October meetings.

Officers of the Southern California District are:

Chairman: E. Tonjes, The Flintkote Co., Los Angeles
Vice-chairman: T. Pringle, Jr., Menasha Container of California, Anaheim
Secretary: D. T. Snell, Continental Can Co., Los Angeles
Treasurer: L. R. Giersch, Crown Zellerbach Corp., Los Angeles
Membership Chairman: S. Feldman, The Flintkote Co., Los Angeles
Publicity Chairman: L. L. Larson, Potlatch Forests, Inc., Pomona

Meetings will be held at the Roger Young Auditorium, 936 West Washington Blvd., Los Angeles, on the second Tuesday of even-numbered months. The technical program will begin at 4 p.m., and the dinner at 6 p.m. Notices of these meetings will be mailed to TAPPI members in the area and to all members of the district.

Meetings held in August and October attracted a wide attendance from mills and converting plants in Southern California. On December 12 the dinner meeting will feature a paper on "Industrial Engineering and Management Problems in the Paper Industry," by Professor W. Whitston of UCLA. All who are interested in papermaking and converting technology are invited to attend this meeting.

*E. Tonjes, The Flintkote Co., chairman Southern California District*

## *Thirteenth Testing Conference*

"Testing for the Graphic Arts" has been selected as the theme of the 13th Testing Conference of the Technical Association of the Paper and Pulp Industry. William R. Willets, Titanium Pigment Corp., New York, N. Y., has been appointed general chairman of the conference, which will take place Sept. 18 to 21, 1962, at the Benjamin Franklin Hotel, Philadelphia, Pa.

Mr. Willets announced that the program chairman is Miss Jacqueline Fetsko, assistant research director of the National Printing Ink Research Institute, Lehigh University, Bethlehem, Pa. John Fachet, executive vice-president of the Thwing-Albert Instrument Co., Philadelphia, Pa., will be in charge of Local Arrangements.

Tentative plans call for the technical sessions to be held on Wednesday and Thursday, Sept. 19 and 20, 1962. Tuesday, September 18, will be devoted to committee meetings of the TAPPI Testing Division and the conference will close on Friday, September 21, with visits to prominent paper and printing establishments in the Delaware Valley. A program for ladies will also be arranged.

**Attention Authors**

**47th Annual Meeting**

**Deadline**
**for**
**Abstracts**
**and**
**Manuscripts**
**January 1, 1962**

## *Sixth Deinking Conference*

The 6th Deinking Conference was held Oct. 4–6, 1961, at the Hotel Harris, in Kalamazoo, Mich. This Deinking Conference was the largest to date, 149 people attending.

The 1st Deinking Conference was also held in Kalamazoo in 1956. While the increased attendance at Deinking Conferences may not be as great numerically as other TAPPI conferences, the interest of the people attending and the quality of the papers continues to rank high.

The conference this year was devoted to papers on the bleaching of deinked stock and the papers were not only well presented, but proved to be of substantial interest because the discussions ran overtime at both sessions.

An outstanding feature of the conference was an address by J. D. Dailey, senior vice-president of Allied Paper Corp., at a banquet sponsored jointly by the 6th Deinking Conference and the Kalamazoo Valley Section of TAPPI, J. D. Chadderdon, Kalamazoo Paper Co., chairman.

R. G. Macdonald, treasurer, TAPPI, presented the Kalamazoo Valley Section with a new banner (to replace one which was lost) and a gavel.

A number of very interesting plant tours was arranged by R. Bennett, Allied Paper Co. On Thursday, a group of 65 visited the Pioneer Paper Stock Co., and Allied, King and Bryant mills; and on Friday, a group of 50 visited the Kalamazoo Paper Co.

A. J. Felton, The Black-Clawson Co., Middletown, Ohio, chairman of the Deinking Committee, was general conference chairman. The technical program chairman was B. C. Sisler, Bergstrom Paper Co., Neenah, Wis., and the local arrangements chairman was S. J. Stetz, Allied Paper Corp., was plant tours chairman.

## *Forty-Seventh Annual Meeting*

The 47th Annual Meeting of the Technical Association of the Pulp and Paper Industry will be held Monday, Feb. 19, 1962, at 10:00 a.m. in the West Ballroom of the Commodore Hotel in New York, N. Y. The reports of TAPPI's President, Harold M. Annis, Oxford Paper Co., New York, N. Y., and of the Executive Secretary, P. E. Nethercut, TAPPI, New York, N. Y., will be presented. Other reports will also be given.

In conjunction with the Annual Meeting there will be a series of 25 technical sessions beginning Monday, Feb. 19, 1962, and concluding Thursday noon, Feb. 22, 1962. The following committees are sponsoring these sessions:

Adhesives Testing Committee. Chairman, R. C. Brown, U. S. Envelope Co., Metuchen, N. J.
Alkaline Pulping Committee. Chairman, D. Wetherhorn, W. C. Davis Associates, Savannah, Ga.
Chemical Products Committee. Chairman, P. B. Borlew, Celulosa de Chihuahua S.A., Chihuahua, Chih, Mexico.
Corrugated Containers Industrial Engineering Committee. Chairman, R. S. Owen, R. S. Owen & Co., Chicago, Ill.
Corrugated Containers Production Committee. Chairman, L. F. Ashwood, Downing Box Co., Milwaukee, Wis.
Cylinder Board Committee. Chairman, C. D. Coffman, Chesapeake Paperboard, Baltimore, Md.
Dissolving Pulps Committee. Chairman, M. A. Heath, Buckeye Cellulose Corp., Memphis, Tenn.
Forest Biology Committee. Chairman, D. D. Stevenson, Buckeye Cellulose Corp., Foley, Fla.
Fourdrinier Committee. Chairman, R. W. Peters, Eastman Kodak Co., Rochester, N. Y.
Fundamental Research Committee. Chairman, R. P. Whitney, The Institute of Paper Chemistry, Appleton, Wis.
Graphic Arts Committee. Chairman, H. J. Connell, S. D. Warren Co., Cumberland Mills, Me.
Operations Research Committee. Chairman, E. F. Thode, The Institute of Paper Chemistry, Appleton, Wis.
Packaging Materials Testing Committee. Chairman, K. W. Ninnemann, Olin Mathieson Chemical Corp., New Haven, Conn.
Pulp Purification Committee. Chairman, R. J. Auchter, Kimberly-Clark Corp., Neenah, Wis.
Structural Fibrous Materials Committee. Chairman, W. H. McPherson, Minnesota & Ontario Paper Co., International Falls, Minn.
Sulfite Pulping Committee. Chairman, P. E. Trout, Waldorf Paper Products, St. Paul, Minn.
Water Committee. Chairman, R. J. Baker, Wallace & Tiernan, Inc., Belleville, N. J.
Wet Strength and Interfiber Bonding Committee. Chairman, C. S. Maxwell, Consultant, Old Greenwich, Conn.

## *Relative Humidity Symposium Proceedings*

"The Proceedings of the 1960 Symposium on Relative Humidity and Paper Test Methods" are now available as a separate report. The 56-page report includes nine symposium papers and discussion on the effect of relative humidity and temperature on the physical properties of paper, the measurement of relative humidity in a test room, and the design and control of test rooms. The symposium was held at the 1960 TAPPI Testing Conference.

Copies of the report may be obtained from TAPPI, 360 Lexington Ave., New York 17, N. Y., at a cost of $1.00 per copy.

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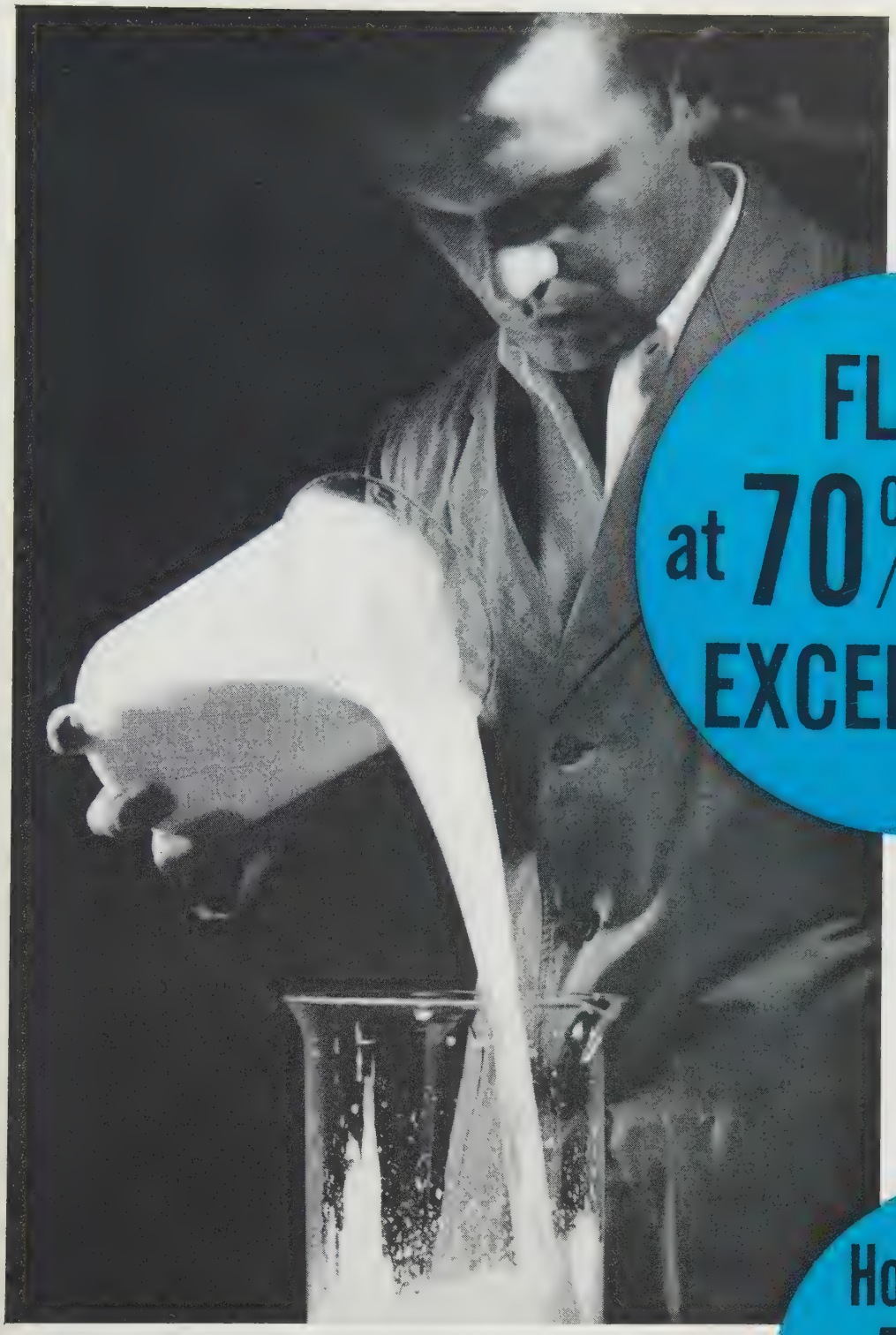

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## *Sixteenth Engineering Conference*

The 16th Engineering Conference, having as its theme "Economic Solutions Through Engineering," was held on Oct. 16–19, 1961, at the Shoreham Hotel, Washington, D. C. Attendance numbered 846 men and 180 ladies.

During the four-day conference period, 73 authors, representing 13 of the Engineering Divisions' 15 committees, presented a total of 54 papers in 16 separate technical sessions. In addition, six panel sessions involving 30 panelists, contributed the weight of their discussions to the overall success of the technical program. The practice of providing preprints, started at the 15th Engineering Conference, was continued with 43 papers being printed, bound, and distributed to those registering for the conference. In addition to the one general and sixteen programmed technical sessions, each of the 15 divisional committees held their own individual meetings after coming together initially on Sunday afternoon, Oct. 15, 1961, for a committee officers meeting.

Among the several highlights of the conference were the keynote address by B. R. Cancell, St. Regis Paper Co., New York, N. Y.; the presentation of Certificates of Appreciation to retiring committee chairmen by TAPPI President Harold Annis, Oxford Paper Co.; and the banquet talk on "The Role of Industry in Space Exploration" given by R. C. Seamans, Jr., Associate Administrator of the National Aeronautics and Space Administration Agency, Washington, D. C., on Wednesday evening, Oct. 18, 1961.

### Keynote Address

Benton R. Cancell, executive vice-president, St. Regis Paper Co., New York, N. Y., delivered the keynote address on Monday morning, Oct. 16, 1961, at the opening session. His remarks complemented the theme of the conference since they concerned the obligations and relationships of engineers to the sales and marketing problems of their respective companies.

The full text of Mr. Cancell's address appears elsewhere in this issue of *Tappi*.

### Certificates of Appreciation

Speaking at the banquet held Wednesday evening, Oct. 18, 1961, TAPPI President Harold Annis presented Certificates of Appreciation to the following retiring committee chairmen:

N. Shoumatoff, The Lyddon Co., London, England, retiring vice-chairman, Engineering Division.
W. A. Moggio, Armstrong Cork Co., Research and Development Center, Lancaster, Pa., retiring chairman, Sanitary Engineering Committee.
H. R. Emery, St. Regis Paper Co., Jacksonville, Fla., retiring chairman, Steam and Power Committee.
John Verwayen, Federal Paperboard Co., Inc., New Haven, Conn., retiring chairman, Drying Committee.
B. C. Kendall, Crown-Zellerbach Corp., San Francisco, Calif., retiring chairman, Industrial Engineering & Materials Handling Committee.
C. A. Lee, Formex Co., Div. of Huyck Corp., Knoxville, Tenn., retiring chairman, Fluid Mechanics Committee.
A. W. Plummer, S. D. Warren Co., Cumberland Mills, Me., retiring chairman, Chemical Engineering Committee.
E. W. Hopper, Consulting Engineer, Pittsburgh, Pa., retiring chairman, Corrosion Committee.
William Pittam, Stone & Webster Engineering Corp., retiring chairman, Construction Engineering Committee.

M. J. Osborne, Bowaters Southern Paper Corp., Calhoun, Tenn., Engineering Division chairman, was also conference chairman. The technical program was arranged by G. E. Shaad, General Electric Co., Schenectady, N. Y., Division secretary. The Ladies' Program Chairman was Mrs. W. S. Allmond, Hopewell, Va. Local arrangements were directed by H. O. Teeple, TAPPI, New York, N. Y.

### Sessions and Papers

In the following meeting account, titles of the papers given, together with those abstracts available at press time are listed below. Preprints are available from TAPPI Headquarters covering the entire collection of papers reported herein, and at a price of $3.00 per copy.

***Certificate of Appreciation Awards: W. A. Moggio, TAPPI President H. M. Annis, B. C. Kendall, H. R. Emery, and E. W. Hopper***

### Session I

The conference was opened on Monday, Oct. 16, at 9:00 a.m., by Engineering Division general chairman, M. J. Osborne. After welcoming those assembled for the 16th Engineering Conference, Mr. Osborne introduced Benton R. Cancell, executive vice-president, St. Regis Paper Co., New York, N. Y., who gave the well received keynote speech.

Following the conclusion of this General Session, the work of the conference began in earnest among the scheduled technical sessions, committee meetings, and panels.

### Session II

The Corrosion Committee held the first of two programmed meetings on Monday, Oct. 16. Titles of the three papers presented during this session were:

"A New Design for a Reinforced Plastic Tank," by F. H. Humphrey, Jr., Metal Cladding, Inc., North Tonawanda, N. Y.

"Inspection of Weld Fabricated Alloy-Clad Digesters," by C. Eldridge, Inspection and Consulting Service, Toronto, Ont., Canada

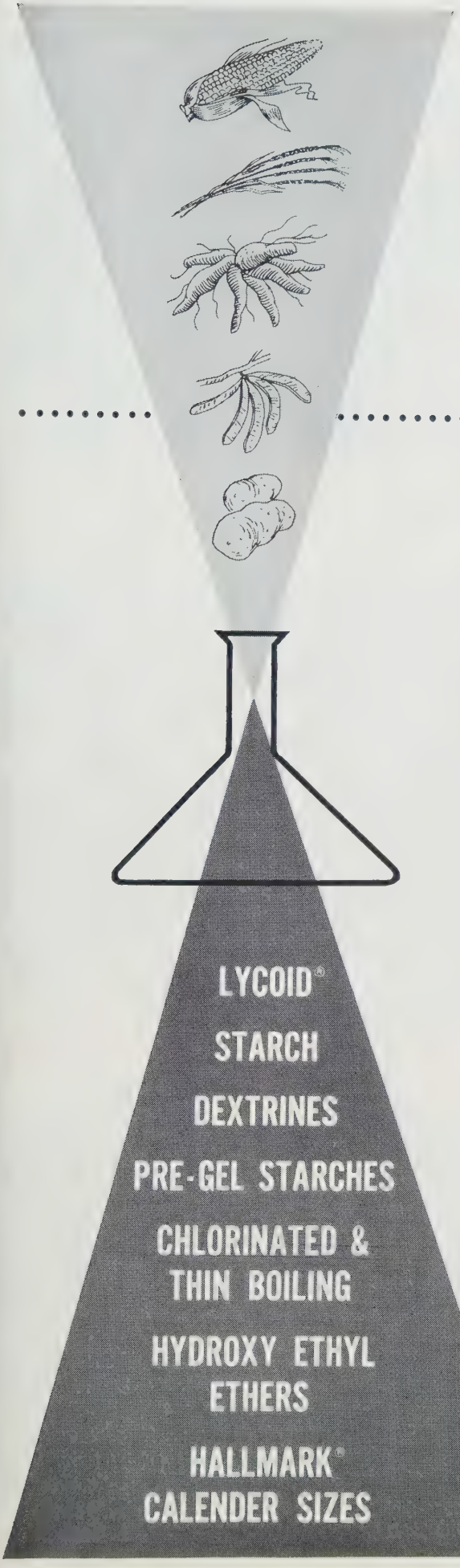
"Successes and Failures in Chlorine Dioxide Manufacturing and Bleach Systems" (C.A 839)—Interim Report, by R. E. L. Wheless, Union Bag-Camp Paper Corp., Franklin, Va.

### Session III

B. C. Kendall, Crown Zellerbach Corp., San Francisco, Calif., was chairman of the third technical session comprising three papers, abstracts of which follow:

"Outside Storage and Handling of Wood Chips on the West Coast," by M. W. Burke, Crown Zellerbach Corp., Antioch, Calif. Outside storage and handling of wood chips has been practiced on the West Coast for over 10 years. Several mills are now in operation which rely completely on purchased chips, with inventories being stored outside for periods as long as 2 to 3 years. Loading, transportation, unloading, and handling procedures are not standardized. Chips are transported by rail car, truck trailer, barge, and air conveyor. Several mills are equipped to receive chips from any one of these sources. Important considerations are compaction obtained when loading transportation equipment, investment requirements, and capacity limitations of the various installations.

"Outside Storage and Handling of Wood Chips in the South," by J. A. Holekamp, American Pulpwood Assn., Atlanta, Ga. This paper presents a collection and further

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*Session 2—Corrosion I: E. W. Hopper, F. H. Humphrey, Jr., C. Eldridge, and R. E. L. Wheless*

*Session 4—Corrosion II: H. B. Harris, Jr., H. M. Canavan, Z. S. Blanchard, and H. Y. Rienhoff*

*Session 3—Industrial Engineering and Materials Handling: B. C. Kendall, M. W. Burke, J. A. Holekamp, and J. R. Girardeau*

*Session 5—Drying: (seated) F. G. Perry, Jr., A. H. Nissan, J. A. Means, and J. A. Villalobos, (standing) S. G. Holt, G. D. King, G. W. Goelz, Jr., and R. Johnson, Jr.*

*Session 4—Corrosion II (Panel): R. C. Stamm, G. E. Linnert, H. Y. Rienhoff, W. E. Henricks, and W. B. Parker*

*Session 6—Sanitary I: W. A. Moggio, A. W. Wilson, R. F. Weston, W. D. Rice, and G. M. Griffith*

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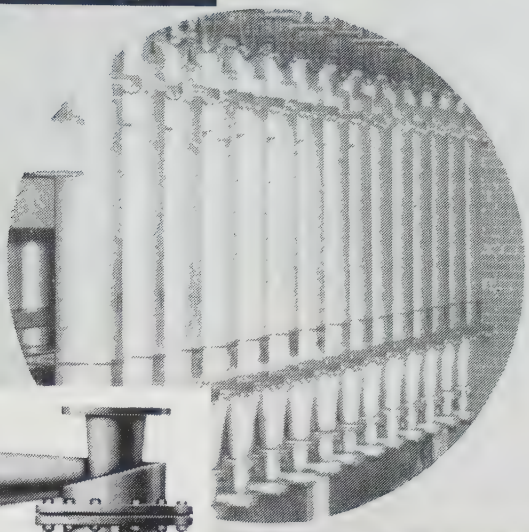

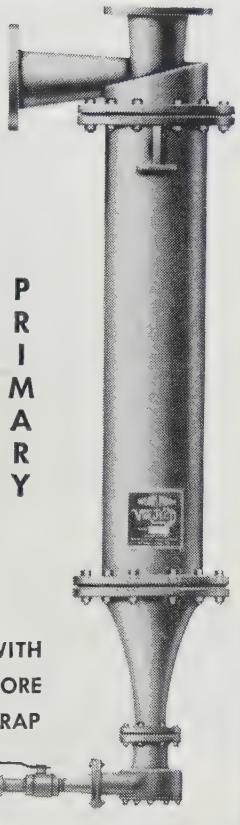

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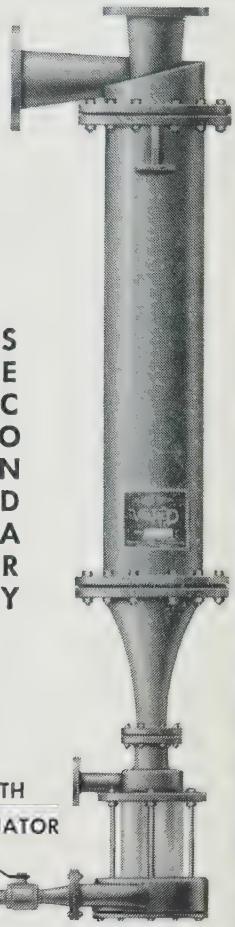

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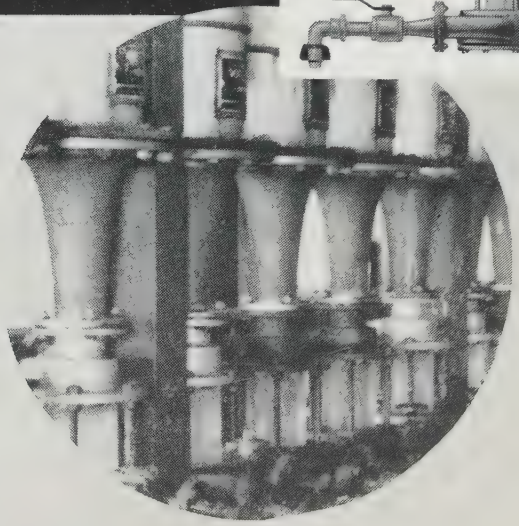

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*Banquet head table: A. S. Landers, G. E. Shaad, H. O. Teeple, and D. D. Hall*

*Banquet head table: J. Lewis, C. C. Wills, E. W. Hopper, and A. W. Plummer*

evaluation of information coming principally from the first four large-scale outside chip storage installations to go into operation in the south. Information has been consolidated into a form which it is hopeful will be thought-provoking, and will encourage further collection of accurate information by those persons considering the merits of outside chip storage. A pulpwood handling, processing, and storage facility at a mill has long been recognized as a vital link between pulp and paper manufacture on one hand and woodlands operation on the other. This link often exercises greater influence over the successful and profitable operation of both than is generally recognized by mill and woodlands management alike. Outside wood storage and handling in chip form promises significant economies as soon as equipment goes into operation. These economies can be calculated and applied to substantial quantities of wood fiber. For the future, chip storage may well offer an open door to further profit-rewarding and rapid-pace progress in pulp and paper manufacture. Chip storage will surely lead foresters and logging engineers toward better, faster, and cheaper ways to grow, harvest, and more closely utilize the forest crop.

"Wood Storage and Handling in Mills," by J. R. Girardeau, Brunswick Pulp & Paper Co., Brunswick, Ga. A close working relationship between the woods division and woodyard of a company is essential. Working as a team, these two departments can play a major role in determining the cost and quality of the finished product. The proper selection and training of personnel is a determining factor in the degree of efficiency obtained in carrying out established programs. Wood handling methods should be designed for maximum efficiency, based on the programs of wood procurement and mill operations. Retention time of trucks in mill yard affects the profit of producers, which in turn is reflected in cost of wood delivered to plant. Selection of yard equipment, designed for minimum delays in changing from a sling to bucket operation, is vital. Major savings can be realized by minimizing the amount of wood handled from storage. The selection of a method to determine accurately the inventory of wood on hand is necessary to control the movement of inbound wood and to determine mill usage.

Session IV

The second Corrosion Session was conducted under the chairmanship of H. B. Harris, Jr., Union Bag-Camp Paper Corp., Savannah, Ga. Available abstracts are given below for the four papers presented at this session as well as the panel discussion which was a part of it.

"Current Status of Alkaline Digestor Corrosion as Reported by Regional Digester Group" (C.A. 522), Interim Report. This is the sixth annual report covering regional digester activity. TAPPI Mill Report Forms were received from 31 mills and included 290 carbon steel, stainless steel, and Inconel clad or lined digesters. The corrosion rates contained in this report correspond closely to that of previous reports. The critical area has changed to the middle shell and overlay welding of carbon steel digesters with stainless steel has increased. The information contained in this report should be of value to pulp mills as a comparison of their digester corrosion problems to that of other mills and with regard to future digester design and maintenance.

"Report of Stainless Steel Overlay Welding Experience in Pulp Mill Digesters," by Paul C. Bobo, Division Engineer, The Mead Corp., Kingsport, Tenn., and Zane S. Blanchard, division manager, Portland Overlay Welding Div., Chicago Bridge & Iron Co., Portland, Me. Overlay welding of kraft and soda digesters has been in general use for a number of years. The results obtained have varied considerably. This experience has ranged from over 10 years' satisfactory service life to failure within 6 months of application. The majority of pulp digester overlays have performed well. However, a great amount of work has been accomplished to determine the cause in those overlays which failed, and in many cases this has been handicapped by lack of detail with regard to application and service. This report is prepared to provide the complete details regarding welding procedures, materials used and service experience in six digesters located at The Mead Corp., Kingsport, Tenn. Records have been maintained of chemical and metallurgical studies and visual inspections of this work. The first overlay work was performed at this mill in early 1958, with additional work being performed in 1959 and 1960. This report should be of assistance to mill personnel who have digester corrosion problems, and to personnel engaged in laboratory study aimed toward improving digester overlay welding techniques.

"Overlay Testing at Time of Application" by Hugh Y. Rienhoff, Alloy Cladding Co., 3649 Keswick Road, Baltimore, Md.

The panel discussion was moderated by R. C. Stamm, West Virginia Pulp & Paper Co., New York, N. Y. An abstract of "Recommendations of Installation of Weld Metal Overlay Linings of Stainless Steel in Sulfate and Soda Pulp Digester Vessels," Corrosion Committee Assignment No. 4039—Interim Report, is given below. This document covers internal linings of stainless steel weld metal deposited as overlay on the surface of pulp digesters to extend the useful life of the vessel. The recommendations herein provide guidance both to the vessel operator and to the welding operator on the essential qualities of a weld overlay lining and the precautions to be observed in its installation. Since this is a pioneering effort to bring about standardization and quality

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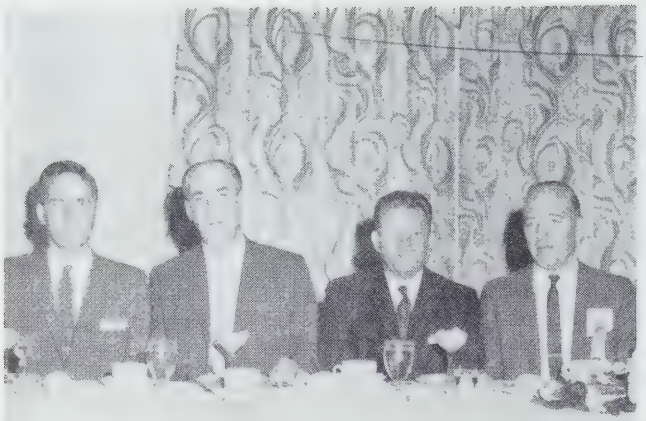
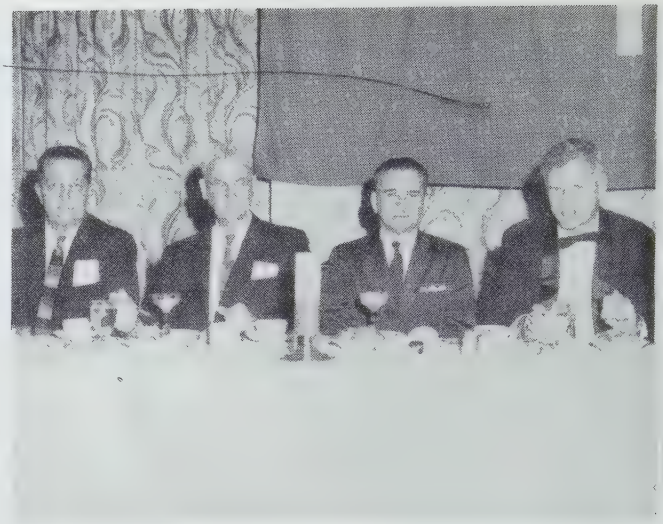


*Banquet head table: H. R. Emery, W. C. Bloomquist, W. A. Moggio, and B. C. Kendall*



*Banquet head table: C. A. Lee, K. O. Elderkin, TAPPI President H. M. Annis, and R. C. Seamans, Jr.*

control on this particular article, the first issue of this recommended practice serves a threehold purpose by pointing out *what* should be required of a newly applied overlay lining, *how* these qualities should be measured on appropriate samples, and *why* these qualities are essential in gaining satisfactory performance from the lining in service. It is anticipated that this document will eventually become the basis for a TAPPI Standard of Recommended Practice after opportunity has been afforded to industry representatives at large to comment on it.

Panelists at this session were as follows:

Specifications: G. E. Linnert, Armco Steel Corp., Baltimore, Md.
Applicators: H. Y. Rienhoff, Alloy Cladding Co., Inc., Baltimore, Md.
Mill owners: W. E. Henricks, The Mead Corp., Chillicothe, Ohio
Insurance companies: W. B. Parker, The Hartford Steam Boiler Inspection and Insurance Co., Hartford, Conn.

### Session V

The fifth technical session was held by the Drying Committee under the chairmanship of F. G. Perry, Jr., Arthur D. Little, Inc., Cambridge, Mass., and included a panel discussion of "High Performance Hoods for Coating Drying," by S. G. Holt, Consolidated Water Power & Paper Co., Wisconsin Rapids, Wis.

Panel members were:

G. D. King, Crown Zellerbach Corp., West Linn, Ore.
G. W. Goelz, Jr., Blandin Paper Co., Grand Rapids, Minn.
R. M. Senkowski, Lee Paper Co., Vicksburg, Mich.
R. Johnson, Jr., Oxford Paper Co., Rumford, Me.

Abstracts and/or titles of the three papers presented at this session are given below.

"Heat Transfer and Water Removal in Cylinder Drying, II. Felted Cylinders," by A. H. Nissan, Rensselaer Polytechnic Institute, Troy, N. Y.; H. H. George, Jr., E. I. du Pont de Nemours & Co., Inc., Richmond, Va.; and D. Hansen, Rensselaer Polytechnic Institute, Troy, N. Y. An earlier theory of the heat and mass transfer on unfelted drying cylinders has been extended to include felted cylinders. Data from an experimental cylinder drier are compared with the theory. An analysis of the experimental drying data is presented in terms of the various mechanisms for water removal on a cylinder drying machine. It was found that the heat transfer through the sheet could be satisfactorily described in terms of conduction alone for cylinder temperatures below the boiling point of water. At higher temperatures convection mechanisms become increasingly more important. The extraction of water by the felt also increased with the cylinder temperature, the increase being especially marked near the normal boiling temperature for water.

"Water Removal by High Velocity Air Hoods," by J. A. Means, Time Inc., Springdale, Conn. Several high velocity drier hood installations were studied using a technique not requiring sheet samples. Drying rates on paper and coating were compared with velocities, temperatures, and B.t.u. inputs.

"The Determination of High Velocity Drier Performance on a Paper Machine," by J. A. Villalobos, Ross Engineering Co., New York, N. Y.

### Session VI

The first of two scheduled meetings of the Sanitary Engineering Committee was held Monday afternoon, October 16. Committee Chairman W. A. Moggio, Armstrong Cork Co., Lancaster, Pa., was also session chairman, and introduced A. W. Wilson, editor, *Pulp & Paper*, New York, N. Y., who addressed the committee meeting as session keynoter. Editor Wilson's remarks represented an appraisal of current official attitudes toward the pulp and paper industry's multimillion dollar effort to eliminate pollution in the nation's streams, and the effect upon this program of the recent enactment of a stronger Federal antipollution law. Abstracts and/titles of the three papers presented are given below.

"A Lagoon System for Treatment of Bleach-Plant Effluent," by C. S. Huestis, Continental Can Co., New York, N. Y.

"New Bio-Treatment Design and Performance—Pulp and Paper Waste," by R. F. Weston, Roy F. Weston, Inc., Newtown Square, Pa.; and W. D. Rice, P. H. Glatfelter Co., Spring Grove, Pa. In cooperation with the clean streams program of the Pennsylvania Sanitary Water Board, the P. H. Glatfelter Co. has provided primary waste treatment and instituted in-plant modifications and good housekeeping procedures. In addition they have recently constructed secondary biological waste treatment facilities. Studies were carried out to establish the treatment requirements and to determine the most feasible secondary treatment procedure. In addition, special studies were made to establish the feasibility of aeration using a turbine near the surface. Preliminary investigations indicated that the Stabilization Basin biological treatment method had the lowest first and operating costs. A $2^1/_2$ m.g.d. demonstration unit was installed to determine the practicability of the treatment method. Changes in the treatability of the waste, inherent deficiencies in the treatment method and a modification of long range plans made it advisable to re-evaluate the other

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*Session 7—Sanitary II: R. W. Simpson, W. A. Moggio, L. L. Klinger, and J. T. Egan*

*Session 10—Process Instrumentation: E. W. Prince, W. A. Wrase, and D. G. Sutherland*

*Session 8—Engineering Economics: G. C. Davis, H. D. Boyce, T. W. Stricklin, F. W. Maiwurm, E. Hooton, Jr., S. D. Srite, Jr., and E. E. Townsend*

*Session 10—Process Instrumentation (Panel): A. W. Plummer, D. L. McGurk, C. W. Carroll, and D. Bates*

*Session 9—Chemical Engineering: J. Lewis, K. I. Mumme, and S. T. Han*

*Session 11—Fluid Mechanics I: P. E. Wrist, S. T. Han, H. Meyer, R. E. White, W. E. Marceau, and J. W. Daily*

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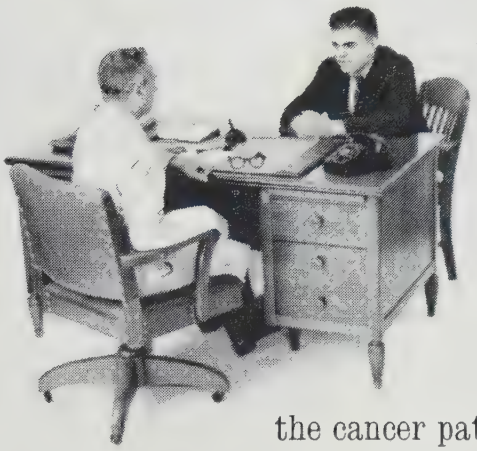

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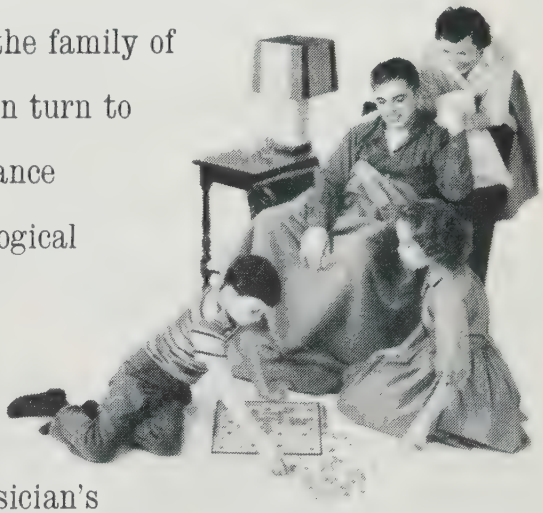


*Banquet head table: M. J. Osborne, Mrs. W. S. Allmond, and P. E. Nethercut*

treatment methods which had been investigated. As a result of the re-evaluation, it was concluded that contact-stabilization activated sludge treatment using surface aerators provided the best treatment procedure for the money. Operating data confirm performance predictions from both batch laboratory biological treatment studies and pilot plant observations.

"An Improved Potomac River—A Case History of a Major Waste Treatment Program," by G. M. Griffith, West Virginia Pulp & Paper Co., Luke, Md.

Session VII

The second portion of the Sanitary Engineering Program was presented October 17 with R. W. Simpson, Gilbert Associates, Inc., Reading, Pa., as chairman. Papers were presented as follows:

"Secondary Waste Treatment at a Specialty Paper Mill," by C. F. Ackerman, Union Mills Paper Mfg. Co., New Hope, Pa.

"Accelerated BOD Removal of Paperboard Wastes," by L. L. Klinger, Whippany Paper Mfg. Co., New Hope, Pa.

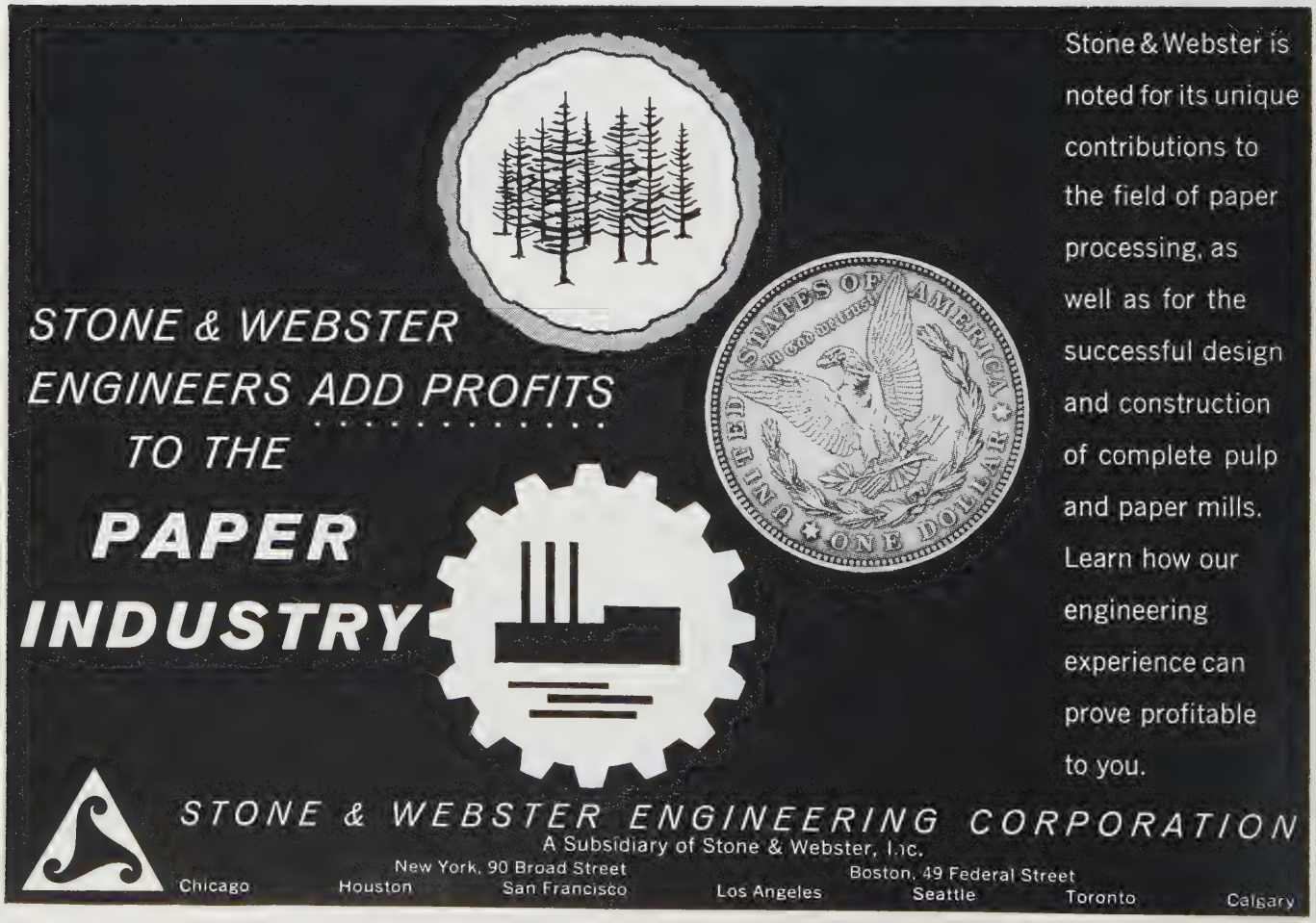
"Plastic Trickling Filters—Design and Operation," by V. A. Minch and J. T. Egan, The Mead Corp., Chillicothe, Ohio; and McD. Sandlin, Rome Kraft Co., Rome, Ga.

Session VIII

Under the chairmanship of G. C. Davis, Bowaters Engineering and Development, Inc., Calhoun, Tenn., the Engineering Economics session presented the following papers.

"Determining the Best Facility for a Company's Needs—from A. "The Owner-User's Views," by T. W. Stricklin, E. I. du Pont de Nemours & Co., Inc., Wilmington, Del. The first step for the owner-user is defining his needs exactly. He communicates these needs—in his own terms—to the designer and builder, and lets them define them in their terms. The owner-user must balance the value of his active participation in the project against the need to give the designer and builder maximum freedom to make their own contributions. He must provide both general direction and free liaison; to do this, he concentrates on defining the problem and any controlling conditions, communicating all pertinent data to designer and builder, and cooperating actively in the solutions of problems as they arise. Throughout the entire project, one objective governs all decisions: the design and construction of a facility which will help the user do his work most effectively.

B. "The Designer's View," by F. W. Maiwurm, E. I. du Pont de Nemours & Co., Inc., Wilmington, Del. A successful facility is designed for its prime function. This calls for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full, first-hand knowledge by the designer of the user's needs. Face to face communication with user and builder during the course of the project will insure that whim is not confused with need, that the structure will be a true expression of design intent, and that the finished facility will be the most economical and efficient possible. Open communications early in the planning stage promotes the maximum use of modular, repetitive design, encourages the application of sound construction practice, and permits more accurate selection of the most promising and economical alternatives.

C. "The Builder's View," by E. Hooton, Jr., E. I. du Pont de Nemours & Co., Inc., Wilmington, Del. Techniques and practices developed in the manufacturing industries are being applied to modern construction with considerable success. As a result, clients may now expect far more than just craftsmanship from builders. Economy and efficiency in the field increase as informed, coordinated planning becomes a part of construction responsibility. Proper advance study and free communication permit the introduction of production line fabrication, enhance application of effective cost controls, and provide a relationship conducive to the prompt recognition and solution of project problems.

"Economic Considerations in the Design of Plant Structures," by S. D. Srite, Jr., and E. E. Townsend, Bowaters Engineering and Development, Inc., Calhoun, Tenn. Economy, in any form, is a welcome to industrial builders. This paper presents some general concepts of economy in the design of plant structures together with three specific examples of economy in design. The three examples presented are the use of fly ash in concrete, prestressed framing for a hardboard mill and prestressed concrete posts for support for pneumatic system.

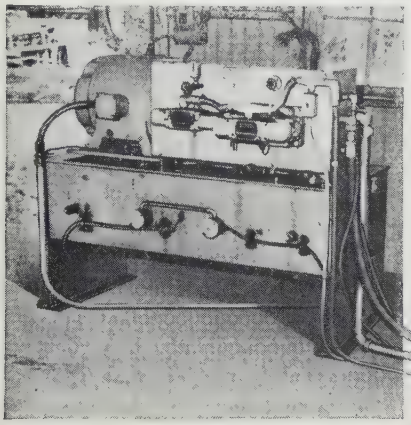
Session IX

The Chemical Engineering Committee meeting was held on Tuesday, October 17, under the chairmanship of J. Lewis,

*Banquet head table: Douglas Jones, executive secretary, Technical Section CPPA, R. G. Macdonald, J. M. Mac Brayne, and S. Andersen*

Pulp and Paper Research Centre, Inc., Lawrence, Mass. Abstracts and/or titles of the papers presented are given below.

"Heat Recovery in Bleacheries," by N. O. Alm, Aktiebolaget Separator Co., Lund, Sweden. In Scandinavia heat recovery in bleacheries has become very common. An investigation of experience has been made in 15 bleacheries to study the different factors that have to be taken into consideration for the construction of the ideal heat recovery system and to choose the most suitable heat exchanger for this purpose. Calories saved, calculated also as fuel oil, and experiences regarding choice of material, frequencies of cleaning, flexibility, etc., are reported.

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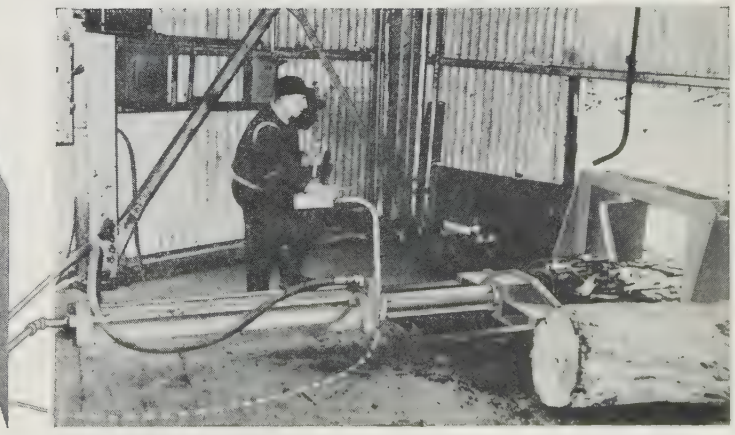

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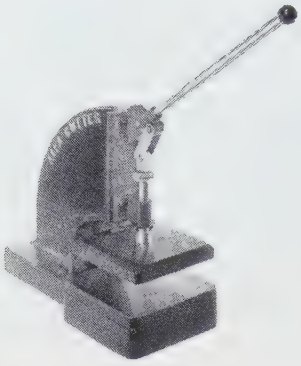

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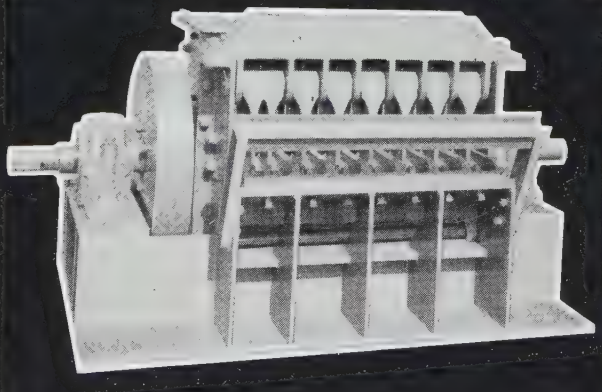

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"Analog Computer Analysis of a Nonlinear Temperature Control System," by K. I. Mummé, Kimberly-Clark Corp., Neenah, Wis. The water temperature in a storage tank was to be maintained by two identical paralleled heat exchangers on the input to the tank. The system configuration and equipment limitations were such as to provide extremely nonlinear system response. The entire system was simulated on an analog computer, where proper design and operation of the systems were discovered. The system contained some interesting features, including a case where, contrary to the usual rules, faster system response resulted in complete uncontrollability. The paper describes the system, the peculiar features of its response characteristics, the boundary conditions imposed by equipment limitations and the ultimate best configuration and operation procedure. Various alternative solutions are also discussed.

"Stock Preparation Analysis," by J. Lewis and D. W. Danforth, Pulp and Paper Research Centre, Inc., Lawrence, Mass. A stock preparation analysis technique is presented in which it is shown that a minimum amount of experimental work is necessary to determine the processing conditions to be used in developing optimum papermaking quality. A brief discussion of basic principles of stock refining is given. In the discussion it is established that applied power and stock throughput are the only two independent stock refining variables after the choice of refining unit and tackle are made. A procedure is described which leads to gathering of a minimum number of samples for freeness tests. The freeness values, when plotted on a stock preparation analysis chart, permit the plotting of a constant freeness line. Three or four samples all at given constant freeness are generated by using different combinations of applied power and stock throughput selected from the constant freeness line on the chart. Quality testing of only the constant freeness samples leads directly to determination of the optimum processing conditions.

Session X

Chairman E. W. Prince, Champion Paper and Fibre Co., Hamilton, Ohio, presided at the Process Instrumentation Committee meeting which included two papers, an interim report and a panel discussion. Abstracts of the papers presented are given below.

"Controlling the Variables," by W. A. Wrase, S. D. Warren Co., Muskegon, Mich. The demand of the customer for a more uniformly superior, high quality product as well as the technological advances in the manufacture of coated printing papers have required the upgrading of manufacturing specifications at the S. D. Warren Co. mill in Muskegon, Mich., to new levels of excellence. To meet these demands it

*Banquet head table: E. W. Prince, E. F. Thode, P. E. Wrist, and A. J. Winchester*

was imperative that steps be taken to gain better control of the variables of manufacture in two basic areas: (1) the treatment of pulps, and (2) the proportioning of pulps, dyes, and fillers to the paper machines. This paper describes the systems evolved and installed, utilizing magnetic flow metering equipment and other control instrumentation, to provide this control. In the area of pulp treatment, through means of accurate measurement and control of flow, consistency and power, all pulps are treated separately to the optimum degree. Again, through the means of accurate measurement and control, these pulps are proportioned with the dyes and fillers, continuously, to four paper machines. After a year and a half of operating experience the following benefits are attributed to this installation: (1) reduced pulp costs, (2) increased pulp production, (3) improved uniformity of paper produced, (4) reduction in manpower, and (5) reduced maintenance costs.

"The Development of an Automatic Freeness Controller," by D. G. SUTHERLAND, The Black-Clawson Co., Middletown, Ohio. A brief history of the development of a continuous freeness controller and a description of the measuring instrument and system for automatically controlling refiners are presented. Instruments now in operation are controlling to less than ±10 ml. C. S. freeness, and have proved to be highly reliable and maintenance-free. Accurate control of freeness has resulted in more uniform paper machine operation, increased uniformity of paper quality and increased production.

An interim report was also given on Committee Assignment 692, "Organization and Importance of the Paper Mill Instrument Department."

The panel discussion related to "Data Handling Systems" and was moderated by A. W. Plummer, S. D. Warren Co., Cumberland Mills, Me.

Panel members were:

T. M. Stout, Thompson Ramo-Wooldridge Computers Co., Los Angeles, Calif.
C. W. Carroll, International Business Machines Corp., Green Bay, Wis.
W. Brickner, The Mead Corp., Chillicothe, Ohio
D. Bates, Potlatch Forests, Inc., Lewiston, Idaho

SESSION XI

C. A. Lee, Formex Co., Div. of Huyck Corp., Knoxville, Tenn., was chairman of the first scheduled session for the Fluid Mechanics Committee.

Titles and/or abstracts of the paper presented are given below:

"The Initial Retention of Fibers by Wire Grids," by R. B. ESTRIDGE, Albermarle Paper Manufacturing Co., Richmond, Va. A more thorough understanding of the mechanism of


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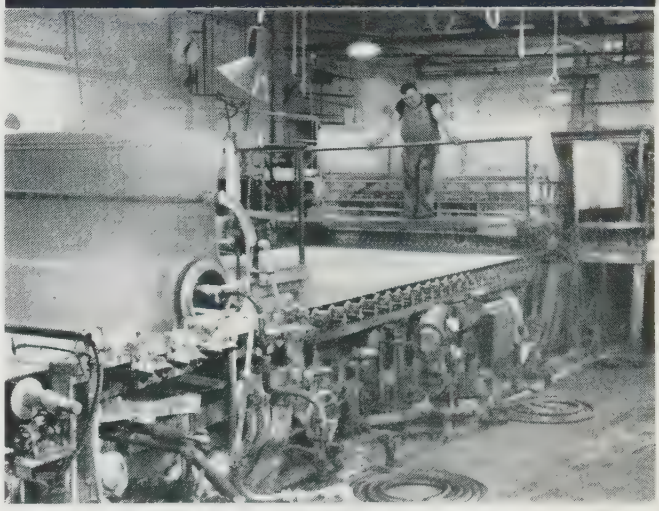

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fiber retention by screens and wire cloth is needed to properly analyze screening and sheet forming operations in the paper industry. Because fibers have extreme axis ratios, the retention of a fiber by an aperture roughly corresponding in size to that of the fiber is a matter of chance. On this basis a general expression predicting the initial rate of retention was developed, and this expression was applied to the simple cases of the initial retention of rigid individual fibers by simple parallel and square-mesh grids. Experimentally determined rates of initial retention agreed with the predicted values for these systems. By comparing results for the two geometries studied, it was shown that the square mesh is most efficient for classifying fibers according to length. For obtaining the greatest retention of fibers during filtration of a fiber suspension, the choice of the best geometry depends upon the relative length of the fibers. The importance of fiber retention in screening and sheet forming is discussed.

"Drainage in a Vacuum Former," by S. T. Han, The Institute of Paper Chemistry, Appleton, Wis.

"A Filtration Theory for Compressible Fibrous Beds Formed from Dilute Suspensions," by H. Meyer, The Mead Corp., Chillicothe, Ohio. A comparison of the experimental results of Ingmanson and Davies with the theoretical results of Happel shows that the constant of D'Arcy's law can, for flow through fibrous mats in the usual ranges of initial consistency and compression, be better described by a multibody drag model than by the capillary model of Kozeny-Carman. Their equations, together with a modified compressibility equation, an empirical retention equation, and equations of conservation of mass, are used to derive general equations of filtration. The nature and experimental determination of the pulp parameters involved in these equations are discussed. Predictions, based on solutions of the equations, for constant pressure and constant rate filtrations are compared favorably with experiment. The application of the theory to papermaking (i.e., to fibrous mats of low basis weight) is discussed and a need is shown for further research into the compressibility of thin mats, and their reaction to pulsating flow and other intermittent disturbances.

"Tne Capillary Behavior of Paper," by R. E. White, Villanova University, Villanova, Pa., and W. E. Marceau, Selas Corp. of America, Dreshertown, Pa. The initial results of research on the capillary behavior of paper, aimed at defining the sheet structure, are presented for consideration and criticism. The problem was attacked from several avenues, including pore size distribution and resistance to the flow of air and water, in the hope of ultimately correlating at least some of the results. Pore size distributions were determined in a capillary tension cell. The results, expressed in terms of effective diameter as a function of water content, indicate that this could be a sensitive discrimination technique for paper. The study of air penetration of wet paper has not yet given the results desired. On the other hand, it has turned up some unexpected behaviors, particularly a strong dependency of flow on time. Water permeabilities, expressed as Darcy coefficients, show little relationship to pore size distribution and are not considered very accurate because of the questionable thickness values. Kozeny coefficients could not be obtained since specific surface values were not known.

"Study of the Flow Characteristics of Dilute Fiber Suspensions: Turbulent Transfer Mechanics with Neutrally Buoyant Suspensions," an Interim Report (PAC 715-PA 192) by J. W. Daily and T. K. Chu, Massachusetts Institute of Technology, Cambridge, Mass.

## Session XII

The Mechanical Engineering Committee session was under the chairmanship of J. S. Mudgett, Strathmore Paper Co., West Springfield, Mass. Seven papers, a motion picture, and a panel session constituted the session program. Abstracts of the papers presented are given below.

"The Design and Mechanical Application of Formex Forming Fabric to the Paper Machine," by J. R. Wagner and J. W. Shearer, Formex Co., Div. of Huyck Corp., Knoxville, Tenn. This paper deals with the design and mechanical application of the Formex forming fabrics to the paper machine. The paper is divided into two parts. The first part discusses the fabric aspects, and the second part discusses the mechanical aspects. In the first section the basic differences between metallic fourdrinier wire and the Formex forming fabric are discussed, giving the physical properties and including operational characteristics and advantages. A new Formex application, that of the drier fabric, is outlined and the basic advantages of this type application is mentioned. The second section discusses the equipment normally used, as well as paper machine changes made possible by the fabric's flexibility and light weight. In this section another new fabric application, the Power Fabric, is discussed and its advantages are also given.

"Application of the Roll-less Flo-Vac on a Fourdrinier Machine," by R. S. Smith, West Virginia Pulp & Paper Co., Covington, Va. The development of the roll-less Flo-Vac and its installation on a fourdrinier manufacturing semi-chemical corrugating medium are described. Results of the installation with regard to wire wear, couch load, and production are listed. A reduction in wire wear of 40 to 50% was effected, which should improve wire life by 80%. Couch load was reduced by 30%. An increase in machine speed of 4% was realized. Indications are that a roll-less Flo-Vac installation should be considered for any fourdrinier machine having wire wear problems. Its use on machines where quality suffers as a result of poor water removal should also be considered.

The panel discussion, moderated by R. T. Depan, the H. K. Ferguson Co., Cleveland, Ohio, presented the following

papers during its discussion of "Mechanical Features of Continuous Digester Systems."

A. "Development of Defibrator Continuous Digesters," by U. Lowgren, American Defibrator, Inc., New York, N. Y. This paper briefly reviews the development of the continuous Asplund defibrator and the evolution of the present day digester with its various components. The application of these digesters in the industry is reviewed with emphasis on the continuous pulping of wood chips for the roofing-flooring industry, insulating and hardboard, semichemical pulping for 9-pt. and bleachable grades, and the employment of the digesters for dispersing asphalt and similar impurities in waste paper for the purpose of reclaiming same. The different kinds of digester designs are reviewed.

B. "The Modern Continuous Digester," by W. Herbert, Pandia Div., The Black-Clawson Co., Middletown, Ohio. The various mechanisms which comprise a modern continuous digester of the horizontal type are described. Various means of metering and feeding the digester are presented. Recent improvements in design of screw feeders and rotary valve feeders are discussed and illustrated. The author explains how the digester proper functions, including means employed to obtain high rates of recirculation of cooking liquor. Design improvements in rotating impeller dischargers are shown, and the application of rotary valves and screw presses as discharge mechanisms discussed. Methods used to control and instrument the digester are given.

C. "The IMPCO Digester" (motion picture), shown by J. K. Perkins, Improved Machinery, Inc., Nashua, N. H.

D. "Continuous Digester Specifications, Applications and Operation," by R. G. Goodwin, Bauer Bros. Co., Springfield, Ohio. The major details of interest to pulp and paper engineers are outlined and discussed for three types of digesters. These are the rapid cycle, Grenco and M & D digesters. These digesters are all high pressure units using working pressures up to 300 p.s.i.g. They are all certified according to the ASME code for unfired pressure vessels. The material common to all three types and used extensively in all cases is type 316 stainless steel. Solid plate is normally used. However, stainless clad has been used in this type of construction. The Grenco valve, which is used with two of these digesters, is a precision built, plug-type design unit. Horsepower requirements are low for this feeding and discharging equipment, generally in the 5 to 10 hp. range. The three digesters cover a wide range of pulp mill applications, particularly in the high yield field. They are now beginning to move into the kraft and bisulfite pulping operations. A series of charts, flow diagrams, and mill photographs are used to illustrate the equipment and outline the material requirements.

E. "Kamyr Continuous Cooking Systems," by N. R. Baldwin, Kamyr, Inc., Hudson Falls, N. Y. Kamyr, Inc., now have in operation or on order approximately 63 units producing $4^1/_2$ million tons of pulp annually. Initial development of the continuous digester was concentrated on the kraft process. The kraft unit is now producing pulp of very superior quality utilizing the diffusion-extraction system. Kamyr recently has been concentrating on developments and modifications to permit continuous pulping utilizing other raw materials and processes. The paper will give a brief description of Kamyr continuous cooking equipment available including the top quality kraft digester, the new corrugating and linerboard digester, the cold caustic pressure impregnation unit, and the new sodium bisulfite digester.

Session XIII

The second session of the Fluid Mechanics Committee was held Wednesday, October 18, with P. E. Wrist, The Mead Corp., Chillicothe, Ohio, as chairman. Titles and/or abstracts of the papers printed are given below.

"On the Instability of Stock on a Fourdrinier Wire," by W. R. Debler and C. S. Yih, College of Engineering, Uni-


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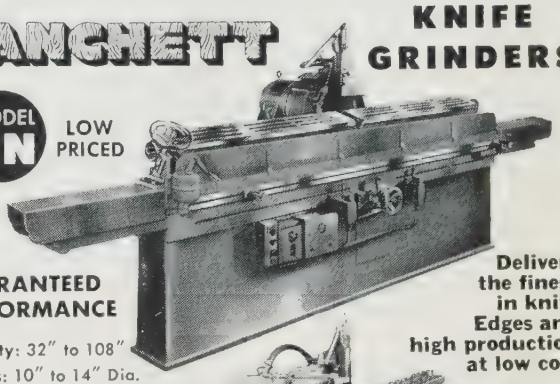

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versity of Michigan, Ann Arbor, Mich. A case of free-surface instability closely related to the phenomenon of spouting on a fourdrinier wire is investigated. As a rotating cylinder with a liquid film attached to its inner surface is slowed down or stopped instantaneously, the liquid will be retarded near the surface of the cylinder while farther away the speed remains high. This is an unstable situation, and is similar to the situation prevailing at a stock-carrying fourdrinier wire as it leaves a table roll. In the latter case the fluid is slowed down at the near-stagnation region on the wire as it leaves the table roll, while the free-surface velocity remains high. The wavelength of the most unstable mode can be obtained from the mathematical analysis that has been carried out up to but not including the numerical stage. In the analysis the effects of Coriolis force and of surface tension are considered. Since previous experience has shown that the role of viscosity is only secondary in the determination of the most unstable mode, it will be taken into consideration only insofar as it affects the velocity of the primary flow, which is unsteady. Experimental data obtained indicate that the critical wave number ($2\ \tau$ times the film thickness divided by the critical wavelength) of the most unstable mode is 2.7 approximately.

"Stability of a Rotating Liquid Film," by C. S. Yih, College of Engineering, University of Michigan, Ann Arbor, Mich. The results of an investigation of the stability of a liquid film attached to the outside of a rotating cylinder are presented here. The problem is pertinent to the growth of disturbances as they pass over the table rolls beneath a fourdrinier wire, and may have some bearing on the problem of patterns formed on the surface of coatings applied by roll coaters, commonly referred to as "film-split patterns." A dimensional analysis and an outline of the mathematical theory are given, and experimental results on the wave number (or wavelength) of the most unstable mode of the disturbance are presented in usable form. The paper is a shortened version of an original paper published in the *Proceedings of the Royal Society of London*, 1960.

"Theoretical Considerations of Vortex Separators," by J. D. Boadway, Consolidated Paper Corp., Ltd., Grand'-Mere, Que. Classification of suspensions can be achieved by means of centrifugal separation made possible through the use of a fluid vortex. This paper treats with the development of such devices, dealing primarily with the theoretical knowledge developed during the progress of the research. Whereas the relationship between flow and pressure drop across the device and the effect of scaling up or down is relatively simple, the effects of change design proportions is more complex. By making simplifying assumptions in regard to the energy loss by fluid shear, a partial understanding can be achieved. The separation of particles can also be understood through use of the same assumptions. The vortex devices contain a gas core in the center. Through an understanding of hydraulic conditions within the device, designs can be made which remove gases from suspensions. The development during which this theoretical understanding has been achieved has led to several devices of commercial significance, among them a unit known under the trademark Vorject—a centrifugal cleaner, and the Vorvac—a device for simultaneous cleaning and gas removal.

"The Design of Manifold Systems for Paper Machine Headboxes," by J. Mardon, D. W. Manson, and J. E. Wilder, Oxford Paper Co., Rumford, Me.

"The Behavior of Two Dimensional Slice Jets," by J. J. Stukel and G. L. Calehuff, West Virginia Pulp and Paper Co., Covington, Va.

## Session XIV

H. R. Emery, Jr., St. Regis Paper Co., Jacksonville, Fla., was chairman of the Steam and Power Session which presented two papers and also included a panel discussion.

P. L. Nelson, Chas. T. Main, Inc., moderated the panel discussion on the subject "Projected Thinking on Design of Paper Mill Power Plants."

Panel members were as follows:

G. L. Yeakel, Gilbert Associates, Inc., Reading, Pa.
J. G. Hoad, John G. Hoad & Associates, Ypsilanti, Mich.
R. E. Baharian, The M. W. Kellogg Co., New York, N. Y.

Abstracts and/or titles of the papers presented at this session are given below.

"Comparisons of One Large Versus Two Small Turbine Generators for Paper Mill Power Plants," by T. J. Judge, International Paper Co., Mobile, Ala.

"Centralized Control of Steam and Electric Power in the Pulp and Paper Mill," by K. L. Fish, Jr., Great Northern Paper Co., Millinocket, Me. The first phase of this paper will acquaint you with the Great Northern Paper Co.'s steam and electric power system and the control that has been built into the system. The Great Northern generates all of its own power with hydroelectric turbines, hydromechanical turbines, thermal reducing turbines, and thermal condensing turbines. Slides are shown illustrating the power system which has a power generation capability of 167.4 Mw. The methods used by the Great Northern for control of this system are covered in this paper. The second phase of this paper covers the high-pressure steam power plant at each mill. The Millinocket power plant was built approximately two years after the East Millinocket plant and incorporated many design improvements. These design improvements offer a perfect opportunity to discuss each power plant and to show the advantages of the centralized control room which was incorporated into the Millinocket power plant. High priority will be given to centralized control at the Great Northern Paper Co. in any future steam and electric power expansion program.

## Session XV

E. F. Thode, The Institute of Paper Chemistry, Appleton, Wis., was chairman of the 15th technical session which presented four technical papers on operations research problems. Abstracts and/or titles of these papers are given below.

"Simulation Models in Operations Research," by E. Naddor, The Johns Hopkins University, Baltimore, Md.

"An Application of Simulation to Carton Inventory," by C. H. Caston, Kimberly-Clark Corp., Neenah, Wis. This paper begins with a brief account of the operations research organization at Kimberly-Clark and its philosophy. We then illustrate one of the applications of O.R. to a study of an inventory problems. A detailed account of an attack on this problem by means of a Monte Carlo simulation is given.

"Resource Allocation and Scheduling for Multiple Projects," by W. Dorfman, C-E-I-R Inc., Arlington, Va.

"The Electronic Computer—It Counts," by F. A. Reed, The Rust Engineering Co., Pittsburgh, Pa.

## Session XVI

S. Andersen, West Virginia Pulp & Paper Co., New York, N. Y., was chairman for the Electrical Engineering Session on Thursday, October 19.

Four final reports and one interim report were presented concerning the status of Electrical Engineering Committee Assignments. Abstracts and/or titles of these reports are given below.

"Proper Application of Motor Control Centers in Paper Mills," a final report on Committee Assignment No. 858, given by Lyman D. Mower, Chas. T. Main, Inc., Boston, Mass. The evaluation of the many ramifications and design features of motor control centers is not included in the scope of this paper. The objective here is to point out certain special application considerations and, in general, evaluate motor control centers from the economic standpoint, particularly as they are applied in the pulp and paper industry.

*Session 12—Mechanical Engineering: (front row) J. S. Mudgett, J. R. Wagner, J. W. Shearer, and W. R. Givin; (back row) R. S. Smith, J. K. Perkins, R. G. Goodwin, and N. R. Baldwin*

*Session 15—Operations Research: E. F. Thode, C. H. Caston, W. Dorfman, and F. A. Reed*

*Session 13—Fluid Mechanics II: P. E. Wrist, W. R. Debler, C. S. Yih, J. D. Boadway, and D. W. Manson*

*Session 16—Electrical Engineering: S. Andersen, L. D. Mower, C. C. Collins, R. J. Minges, E. C. Benjamin, J. V. Cundelan, and L. W. Porter*

*Session 14—Steam and Power: H. R. Emery, Jr., T. J. Judge, K. L. Fish, Jr., P. L. Suter, P. L. Nelson, G. L. Yeakel, and R. E. Baharian*

*Session 17—Maintenance Engineering: R. S. Welch, C. V. Clarke, R. W. Kent, J. J. Yirak, H. W. Smith, L. L. Smith, and C. J. van Riper*

Application considerations with respect to incoming feeder terminations, short circuit coordination, control wiring and cases where motor control centers are not practical have been discussed. Initial installed costs of motor control centers will, in general, be greater than individual field assembled starters. However, considering the saving in building cost, the overall cost differential becomes insignificantly small. In addition, after weighing several other less direct economic advantages plus the various operational advantages, motor control centers can usually be justified economically.

"Power Requirements of Tissue Machines," a final report on Committee Assignment No. 622, given by C. C. Collins, General Electric Co., Schenectady, N. Y. In 1950 TAPPI published data on power requirements for tissue machines. Since 1950 machine design and operational practices have changed sufficiently to warrant re-examination of this published data by the TAPPI Electrical Engineering Committee. During the past two years, operating data on tissue machines has been collected and analyzed. As a result of this engineering analysis new NRL (normal running load) and RDC (recommended drive constant) power constants have been assigned to each driven roll on tissue machines. This report gives the new recommended TAPPI power constants for tissue machines and gives detailed reasons for the selection of each constant. The information contained in this report will be processed later by TAPPI as a Technical Information Sheet.

"Comparison of Types of Reel Drives for Converting Equipment," a final report on Committee Assignment No. 891, given by R. J. Minges, The Gardner Division, Diamond National Corp. In the past the Electrical Engineering Committee of TAPPI has confined itself primarily with the problems of the pulp and paper mills, with little consideration to problems of the converter. Since the converter is the ultimate consumer of our products, and more and more of our companies are becoming converters themselves, either through expansion or acquisition, our committee has set up a subcommittee for "Finishing and Converting Drive Requirements." This is the first in what we hope to be a series of papers concentrating on the electrical problems of the converter. Owing to the extremely competitive market in the converting field it is of the utmost importance that sound judgment be used in the specification of drive requirements for converting equipment. With the limited engineering facilities available to the majority of the converters, they have been placed in the position of depending almost entirely on the equipment and electrical manufacturers, to specify their requirements. While no reflection is meant to be cast on these manufacturers, only too frequently equipment is furnished which is either overdesigned or underdesigned for the particular application. Manufacturers are often bidding on a job where specifications have been so loosely defined that bids are actually not being taken on a competitive basis. It is the purpose of this paper to point out the various types of reel drives available to the converter, along with the various control functions. It is hoped this will give the converter a guide to follow in specifying reel drives so he will be able to arrive at a drive that is economical, functional, and above all provide the characteristics needed for his operation.

"Grounding for Safety in Pulp and Paper Mills," a final report on Committee Assignment No. 4015, given by E. C. Benjamin, M. W. Kellogg Co., New York, N. Y., and J. V. Cundelan, Rayonier, Inc., New York, N. Y. There is a wide variation in recommended grounding practices for the paper industry. In the hope of clarifying the situation, it is the purpose of this paper to review, briefly, the various aspects of the problem as they occur in our industry. It is, of course, impossible to cover exhaustively all phases of so broad a subject in a single paper of reasonable length. Therefore, our attention will be directed toward those situations most commonly encountered and will include suggested procedures for the protection of personnel, equipment, and plant. System grounding has been treated in previous articles and papers. It is not the intention of the authors, therefore, to become involved in this perennial discussion, hence this specific feature will not be considered in this presentation.

"Mill Experience with Sealed Insulation Systems," an Interim Report on Committee Assignment No. 866 was given by L. W. Porter, St. Joe Paper Co., Port St. Joe, Fla.

### Session XVII

H. M. Reed, Jr., International Paper Co., Mobile, Ala., was chairman for the Maintenance Engineering Session which presented two papers, a panel discussion, and a final report on Committee Assignment 4000, "Maintenance Practices Survey." Abstracts and/or titles of the papers presented as well as details of the panel discussion follow.

"Maintenance Measurement and Control," by C. V. Clarke, H. B. Maynard and Co., Inc., New York, N. Y.

"Mill Experience with Dissolving Tank Operation," an Interim Report on Committee Assignment No. 4037, by R. W. Kent, Champion Paper & Fibre Co., Hamilton, Ohio, and J. J. Yirak, Union Bag-Camp Paper Corp., Savannah, Ga.

The panel discussion, having as its subject a final report on Committee Assignment 4000, "Maintenance Practices Survey," was moderated by H. W. Smith, St. Regis Paper Co., Pensacola, Fla.

Panel members were:

L. L. Smith, Bowaters Southern Paper Corp., Calhoun, Tenn.
R. S. Welch, St. Regis Paper Co., Pensacola, Fla.
R. W. Kent, Champion Paper & Fibre Co., Hamilton, Ohio
C. J. van Riper, Union Bag-Camp Paper Co., Franklin, Va.

An abstract of the final report on Committee Assignment 4000 is given herewith. Maintenance costs too frequently have been regarded as a necessary evil and cut percentagewise "across the board" in many so-called cost reduction programs of the past. New appraisals, however, more correctly place efficient maintenance as an integral factor in operating quality, quantity and cost, and a little fact-gathering bearing on the economic aspects of existing maintenance practices seemed to be in order. The purpose of this project, then, was twofold: (1) to secure a cross-sectional viewpoint of existing maintenance practices in the areas of organization, activities and performance of maintenance people, and (2) to secure opinions and suggestions from the respondents on measures which might serve to increase the percentage of maintenance efficiency per man. A questionnaire was prepared and submitted to TAPPI New York Office for distribution to a necessarily limited, but representative, group which had signified a willingness to participate anonymously. Fifty excellent responses were received, and it is regrettable space does not permit reprinting each in its entirety. The following report is a summary of the facts made available through the questionnaire, while comments and opinions expressed by the respondents are indicated as such.

### Seventeenth TAPPI Engineering Conference

The Seventeenth Engineering Conference will be held Oct. 14–19, 1962, at the Queen Elizabeth Hotel, Montreal, Que.

It will be jointly sponsored by TAPPI and the Technical Section, Canadian Pulp and Paper Association.

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# TAPPI NOTES

## *Highlights of the October Executive Committee Meeting*

The Executive Committee voted unanimously to award the TAPPI Gold Medal to **W. B. Campbell** and **C. E. Libby.** These presentations will be made at the TAPPI Annual Meeting to be held in New York next February, and brief biographical sketches of the two recipients will be published in the January issue. Prior to his retirement in 1954 W. B. Campbell was director of Technical Research at the Pulp and Paper Research Institute of Canada, and he has been active since that time as a consultant. C. E. Libby is well known for his many years as an educator at the New York State College of Forestry and the North Carolina State College of Agriculture and Engineering. He is living in Florida now and serving as editor of the "College Textbook of Pulp and Paper Manufacture."

The Executive Committee approved a petition received from TAPPI members in the Los Angeles area wishing to organize the Southern California District of the Golden Gate Section. This petition had been approved by the Golden Gate Section and the Local Section Operations Committee prior to its submission to the Executive Committee. The new district was organized during the summer and has had two dinner meetings this fall. Ernst Tonjes, Flintkote Co., Los Angeles, is the district chairman.

A report of the TAPPI Research Policy Advisory Committee was presented. This committee had been appointed to study TAPPI's responsibilities in the area of basic research. The reports and recommendations of this committee will be published in greater detail after further discussions on the feasibility of establishing a broad program of basic research sponsored by funds from the industry.

The publication and ownership of the "College Textbook of Pulp and Paper Manufacture" will be transferred from TAPPI to the Joint Textbook Committee. This committee is responsible for financing, editing, and revising the four-volume McGraw-Hill Series on "Pulp and Paper Manufacture." Members of the Joint Textbook Committee are H. S. Spencer, chairman, F. P. Doane, Jr., A. C. Hill, D. Jones, and R. G. Macdonald. The 1960 edition of the "College Textbook" is being revised by C. E. Libby, editor, and a number of authors of individual chapters. The revision is scheduled for publication between hard covers in late summer of 1962, so that it will be available for use in the pulp and paper colleges at the start of their 1962–1963 year.

An additional allocation has been made to the Project Appropriations Committee for its use in the October review of committee proposals requiring financial support. This brings to roughly $63,000 the TAPPI funds available to assist committees in their authorized projects this year.

The establishment of a TAPPI Student Loan Fund to assist needy students preparing for technical careers in our industry has been deferred. A recent survey of the pulp and paper schools indicated that most already have adequate scholarships and loan funds available for their students.

The Executive Committee endorsed a proposed amendment of the TAPPI Constitution on the balloting requirements for Association officers and incoming Executive Committee members. If approved by the voting membership of the Association, the amendment would permit the election by acclamation of those positions for which only one candidate has been nominated. This amendment would not alter the present provisions for nominating several candidates for a single office, but it would obviate the need for a letter ballot to all voting members when only a single slate of candidates is nominated. The official notice of this proposal will be mailed shortly to all voting members of the Association, and the subject will be placed on the agenda for the Annual Business Meeting to be held in New York in February.

A request for TAPPI financial support of the 1962 activities of the Paper Industry Career Guidance Committee was approved. This committee includes representatives of TAPPI, APPA, PIMA, NPA, and NPTA, and its programs are financed jointly by these five associations. This committee has been in operation for two years, and its 1962 program includes the employment of a full-time counselor to coordinate all career guidance efforts of the five associations.

The Executive Committee discussed the feasibility of a charter flight of TAPPI members to visit pulp and paper mills in Finland next year. It concluded that no action should be taken at this time.

H. F. Arledter, R. A. Dando, R. W. Hisey, E. J. Justus, D. Macaulay, R. H. Neisel, W. C. Walker, and R. S. Williams were named to three-year terms on the *Tappi* Editorial Board.

Philip E. Nethercut
*Executive Secretary*

# TAPPI PROFILE

*Harry F. Lewis*

HARRY F. LEWIS, chairman of the Research and Development Division of TAPPI is Vice-President and Dean Emeritus of The Institute of Paper Chemistry in Appleton, Wis.

He was born in 1891 in Denver, Colo., where his father was a secretary in the YMCA. Home became, successively, Pueblo, Colo., Brooklyn, and finally Manhattan. Public School 6 and the DeWitt Clinton High School in New York City prepared him for Wesleyan University, Middletown, Conn. where he received a B.S. in 1912 and M.S. in 1913. His graduate work in chemistry at the University of Illinois led to a Doctorate of Philosophy degree in 1916. He is a member of Phi Beta Kappa and Sigma Xi.

From 1916 until joining the staff of The Institute of Paper Chemistry in June 1930, he was on the research staff of the Color Investigation Laboratory, USDA Bureau of Chemistry in Washington; in the research department of the National Aniline and Chemical Co., Buffalo, N. Y.; and taught of organic chemistry at Grinnell and Cornell Colleges in Iowa, at Ohio Wesleyan University, and at the University of Maine where, in 1917–18, he gave a course in cellulose chemistry.

Shortly after The Institute of Paper Chemistry was opened in 1930, he was invited by Henry Wriston, President of Lawrence College and Director of the Institute, to join its staff as Professor of Organic Chemistry. At that time the staff of the Institute consisted of Otto Kress, Technical Director, and two students—Harold Bialkowsky and Roy Davis; courses were also given by five Lawrence College professors. From 1933 to 1956 he served the Institute as Dean and Research Associate. In 1956 he retired as Dean and became Vice-President. In September of that year he retired again from the vice-presidency of the Institute and assumed the emeritus relationship; he hopes to be useful to the Institute in this capacity for many more years.

Harry Lewis joined TAPPI early in 1931, and has been a member ever since. In June, 1933, he was asked by Carl Curran, then chairman of the Research and Development Division, to organize and serve as chairman of a new committee to be known as the Fundamental Research Committee. Twenty-five years later he left this post to become chairman of the Research and Development Division. In those twenty-five years the Fundamental Research Committee had sponsored a wide variety of fall conferences either alone or with the related committee of CPPA. These conferences included three lignin roundtables, an international pulp and paper symposium, and symposia on pulpwood resources, wood components, fundamentals of the paper machine, the fundamentals of bleaching, etc. Research projects were sponsored by the committee at various colleges and universities, three TAPPI Monographs have been published, and research papers and symposia were given at many annual meetings of TAPPI.

He was a member of the Executive Committee from 1952–54. In 1955 he was given the Mitscherlich Award of the German Pulp and Paper Association, in 1956 the TAPPI Gold Medal, and in 1959 the American Chemical Society honored him with the Scientific Apparatus Makers Award in Chemical Education.

Perhaps one of his most outstanding contributions to the paper industry has been the zeal with which he undertook the task of making college students aware of the opportunities that a career in that industry could offer. For many years, particularly in the 30's and 40's, he was a touring speaker for the American Chemical Society, speaking on the subject of basic research and the pulp and paper industry to many of the sections of that Society. On other trips he spoke before ACS student affiliate groups and Tau Beta Pi chapters. In all, he has visited more than 200 colleges and universities in the interest of The Institute of Paper Chemistry and the pulp and paper industry. More recently he has been a Visiting Scientist on the program sponsored by the National Science Foundation and carried out by the Division of Chemical Education of the ACS. This program took him to 18 or 20 colleges. On these college and university visits he has stressed the chemistry of wood and opportunities in the paper industry with particular reference to the graduate program of The Institute of Paper Chemistry. When speaking before student groups, Dr. Lewis received valuable help from the Institute alumni group who appeared with him on panels and gave substance to what he had to say about opportunities in the industry.

He has been a member of the American Chemical Society since 1922. In 1934–35, he was chairman of

the Division of Cellulose Chemistry, and in 1955–56 chairman of the Division of Chemical Education. For five years he was a Councilor of the Society. He now represents the Division of Chemical Education on the AAAS Cooperative Committee on the Teaching of Science.

He has published in the Technical Section of the *Paper Trade Journal* and in *Tappi*, in collaboration with his colleagues, on a variety of subjects, including permanence, wood analysis, fiber structure, bark, redwood and its chemical and physical components, lignin, and wood waste utilization; in recent years his publications have been largely in the field of manpower shortages and chemical education.

In addition to the foregoing activities, Dr. Lewis has served on the National Research Council's Committee on the Chemistry of Plant Products (1947–57) and the Committee on Design, Construction, and Equipment of Laboratories (1947–57). He has also been a member of the Pulp, Paper, and Board Division of the International Union of Pure and Applied Chemistry (1951–59), and the Technical Committee on Wood Chemistry of the Food and Agricultural Organization of the United Nations (1946–56). In 1953 he was a United States delegate to the Council of the International Union of Pure and Applied Chemistry, Stockholm.

He has been active in the YMCA on local, area, and national levels, serving the Appleton Association as board member and president, the North Central Area as president, and the National Council as a member for one term. In 1953 he was a delegate to the World Alliance of YMCA's in Geneva, Switzerland. He has been a member of the National Committee on Youth Program for many years.

The Lewis's have 5 children; all are married, and there are 13 grandchildren.


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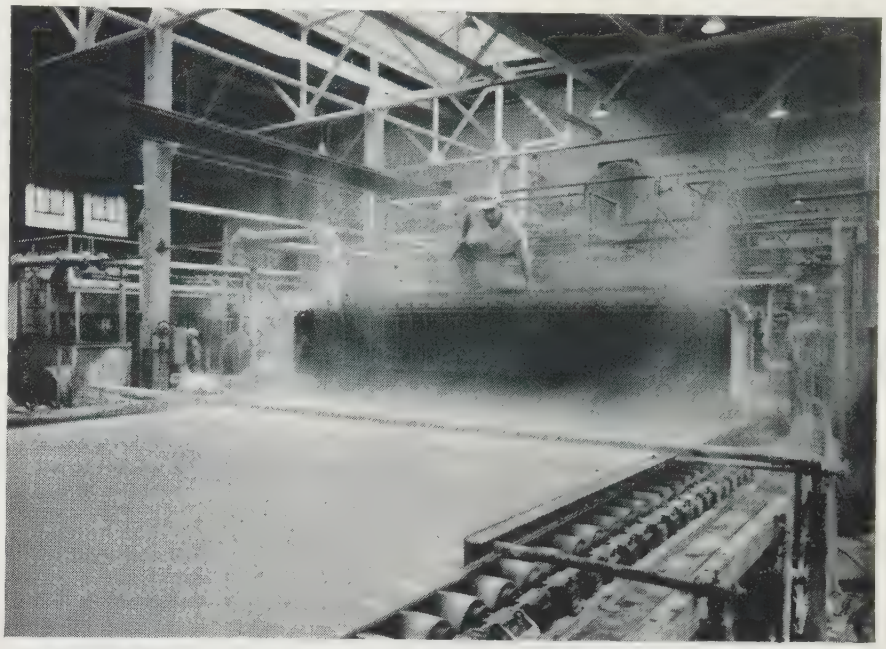

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# PERSONAL MENTION

## *New TAPPI Members*

**Lewis M. Bartlett,** Assistant Groundwood Superintendent, International Paper Co., Corinth, N. Y.

**James C. Bertch,** Salesman, R. T. Vanderbilt Co., Inc., New York, N. Y., a 1958 graduate of Western Michigan University.

**Dwarka Prasad Bhargava,** Graduate Student, New York State College of Forestry, Syracuse, N. Y., a 1953 graduate of Agra College, Agra, India.

**John K. Bonnell,** Sales Representative, Penick & Ford Ltd., Inc., Cedar Rapids, Iowa, a 1949 graduate of University of Iowa.

**Robert H. Buckman,** Assistant to the President, Buckman Laboratories, Inc., Memphis, Tenn., a 1959 graduate of Purdue University.

**William S. Bunte,** Sales Engineer, Frank W. Egan & Co., Somerville, N. J., a 1944 graduate of Rice Institute.

**Kenneth E. Dempsky,** Technical Service and Salesman, Hercules Powder Co., Wilmington, Del., a 1961 graduate of University of Wisconsin.

**Laurence T. Durfee,** District Sales Manager, Peter Cooper Corp., Gowanda, N. Y., a 1936 graduate of New Bedford Institute of Technology.

**Dean W. Einspahr,** Forest Genetics Research, The Institute of Paper Chemistry, Appleton, Wis., a 1949 graduate of Iowa State University with a Ph.D. degree.

**Carl L. Elmore,** Process Engineer, Owens-Illinois Glass Co., Jacksonville, Fla., a 1960 graduate of University of Florida.

**Robert Eykel,** Assistant Manager, Cats-Neparofa, Rotterdam, Holland.

**Ernest Frank,** Supervisor, Customer Service-Research, American Maize-Products Co., Chicago, Ill., a 1946 graduate of University of Chicago.

**Irving S. Goldstein,** Manager, Wood Chemistry Section, Koppers Co., Inc., Monroeville, Pa., a 1941 graduate of Rensselaer Polytechnic Institute with a Ph.D. degree in 1948.

**Holly G. Haworth,** Paper Chemist, Bestwall Gypsum Co., Paoli, Pa., a 1948 graduate of University of Missouri.

**Irving E. Holls,** Assistant Vice-President, Upson Co., Lockport, N. Y., a 1938 graduate of University of Buffalo.

**Francis X. Lee, Jr.,** Sales Engineer, Frank W. Egan & Co., Somerville, N. J., a 1953 graduate of Stevens Institute of Technology.

**Robert F. Lucke,** Development Engineer, Enjay Chemical Co., Div. of Humble Oil & Refining Co., Elizabeth, N. J., a 1952 graduate of Pennsylvania State College.

**Luke L. Lukowiak,** Chemical Engineer, Firestone Plastics Co., Pottstown, Pa., a 1950 graduate of University of Scranton.

**Clayton E. Matthews,** Research Chemist, Arthur H. Thomas Co., Philadelphia, Pa., a 1940 graduate of Philadelphia College of Pharmacy & Science.

**Frank J. Meehan,** Sales Engineer, Frank W. Egan & Co., Somerville, N. J., a 1959 graduate of Newark College of Engineering.

**Henry C. Miller,** Technical Sales Representative, Hooker Chemicals, Ltd., North Vancouver, B. C., Canada, a graduate of University of British Columbia.

**Robert G. Miller,** Sales Engineer, Bird Machine Co., Atlanta, Ga. Attended University of Massachusetts.

**Richard Mollison,** Superintendent, Sonoco de Mexico, S.A., Mexico, D. F., Mexico, a 1958 graduate of University of Texas.

**Hisanori Mori,** President, Hokkai Paper Mfg. Co., Ltd., Otaru, Japan, a 1918 graduate of Keio Gijuku University.

**William S. Morris,** Development Dept., Hillenbrand Industries, Inc., Batesville, Ind., a 1943 graduate of Geo. Williams University.

**William H. Morrow, Jr.,** Marketing Manager of Control Systems for Paper Industry, International Business Machines Corp., New York, N. Y., a 1950 graduate of Georgia Institute of Technology.

**Francis E. Murray,** Research Coordinator, Consolidated Paper Corp., Ltd., Grand'Mere, Que., Canada, a 1950 graduate of University of Alberta, with a Ph.D. degree in 1953 from McGill University.

**Frank Navratil,** Sales & Service Engineer, Bingham Pump Co., New York, N. Y., a 1949 graduate of Newark College of Engineering.

**Harry J. Nossek,** Bacteriologist, R. T. Vanderbilt Co., Inc., East Norwalk, Conn., a 1959 graduate of the University of Connecticut.

**Robert K. Oakley,** Project Engineer, General Services Administration, Washington, D. C. Attended College City of New York.

**Oji Odo Okereke,** Industrial Research, Federal Institute of Industrial Research, Ikeja Airport, Nigeria, a 1960 graduate of University College Ibadan.

**Ermanno Ottino,** Manager, Compagnia Generale di Elettricata, Milano, Italy. Attended Torino University.

**D. J. Lane Pendleton,** Chief Mill Chemist, Albert E. Reed and Co., Ltd., Kent, England, a 1951 graduate of University of London.

**Richard B. Peppler,** Assistant Director of Research, Master Builders Co., Cleveland Heights, Ohio, a 1943 graduate of Ohio Wesleyan University.

**Neftali Puentes,** Process Engineer, Kimberly-Clark Corp., Neenah, Wis., a 1954 graduate of Universidad de Santander.

**M. Dorothy Robinson,** Librarian, Kordite Co., Div. of National Distillers & Chemical Corp., Macedon, N. Y.

**Choi Wha Shik,** President, Hankuk Special Paper Mfg. Co., Ltd., Seoul, Korea, a 1943 graduate of Sensu College, Tokyo.

**Idair I. Smookler,** Librarian, Union Carbide Chemicals Co., So. Charleston, W. Va., a 1939 graduate of University of Delaware.

**Ernest A. Sowell,** Chemist, Anheuser-Busch, Inc., St. Louis, Mo., a 1939 graduate of University of Missouri.

**Geoffrey F. Stonehouse,** Petroleum Technologist, Shell International Petroleum Co., Ltd., London, England, a 1954 graduate of Durham University.

**Fernando Solis T.,** Technical Assistant to Paper Machine Superintendent, Fabricas De Papel Tuxtepec, S.A., Mexico, D. F., Mexico, a 1954 graduate of Escuela Sup. De Ing. Mec. Y Elect.

**Jon Daniel Techentin,** Sales-Service, Hercules Powder Co., Milwaukee, Wis., a 1956 graduate of University of Illinois.

**James H. Watson,** Division Power Engineer, International Paper Co., Mobile, Ala., a 1947 graduate of Georgia Institute of Technology.

**Melville L. Wolfrom,** Research Professor of Chemistry

Ohio State University, Columbus, Ohio, a 1924 graduate of Ohio State University with a Ph.D. degree in 1927 from Northwestern University.

## *Appointments*

**George G. Acheson,** formerly of Milprint, Inc. is now Technical Director of Curwood, Inc., New London, Wis.

**John P. Bainbridge** is now New England Sales Manager for Penick & Ford Ltd., Inc., New York, N. Y.

**Charles Bartell,** formerly of Dewey & Almy Division is now Director of Research and Development for Mystik Adhesive Products, Inc., Northfield, Ill.

**J. Benjamin Beck,** formerly of East Texas Pulp & Paper Co. is now Plant Manager for Cia. Industrial Commercial Del Sur S. A., Montevideo, Uruguay.

**DeWane A. Brenner,** formerly of Simpson-Lee Paper Co. is now a project engineer with Fitchburg Paper Co., Fitchburg, Mass.

**Thomas R. Brossard,** formerly of Boloit Iron Works is now Assistant Sales Manager for Beloit International Corp., C. A., Zurich, Switzerland.

**Albert V. Brucato,** formerly of Fiber Products Research Center, Inc. is now a chemist with Hollingsworth & Vose Co., East Walpole, Mass.

**Bror A. Bygden,** formerly of Anjala Paper Mills is now Vice-President of Tampella A/B, Ingerois, Finland.

**Willard E. Carson** is now Associate Manager of Research, St. Regis Technical Center, W. Nyack, N. Y.

**Cecil N. Carter,** formerly student at North Carolina State College is now a student at The Institute of Paper Chemistry, Appleton, Wis.

**Preston A. Christjohn, Jr.** of West Virginia Pulp and Paper Co. has been transferred from Covington, Va. to New York, N. Y. as Customer Service Engineer.

**David C. Clemens,** formerly of Horkey-Moore Associates is now Director of Valley Fiber, Inc., Wasco, Calif.

**C. William Converse,** formerly of Lyddon & Co. is now Director of the Pulp and Paper Division of the Lummus Co., Paris, France.

**John R. Dam** of the Glidden Co. has been transferred from Baltimore, Md. to Cleveland, Ohio as Director of the Paper Applications Laboratory at the Adrian Joyce Works.

**Louis A. Dela Grange,** formerly of Hamilton Paper Co. is now a sales engineer for the Black-Clawson Co., Watertown, N. Y.

**John G. De Pagter,** formerly of the Crossett Co. is now a process engineer for the Monsanto Chemical Co., Columbia, Tenn.

**Leon R. Eeman,** formerly of MacMillan & Bloedel, Ltd. is now Chief Chemist for Southwest Forest Industries, Inc., Snowflake, Ariz.

**D. Robert Erickson** is now a consulting chemist for Erickson Research Laboratories, So. Laguna, Calif.

**James F. Fenske** of Allis-Chalmers Mfg. Co. has been transferred from Norwood, Ohio to San Francisco, Calif. as Manager of Motor Sales.

**Robert B. Goodman, Jr.,** formerly of Cornell Paperboard Products Co. is now a development chemical engineer with Dymo Industries, Inc., Berkeley, Calif.

**Frederick C. Goodwill,** formerly of St. Regis Paper Co. is now a consultant with Beloit Iron Works, Beloit, Wis.

**William Gutzwiller,** formerly of the Aetna Paper Co. is now Instructor in Paper Technology at Miami University, Oxford, Ohio.

**Robert T. Hart,** formerly of the Gorham Laboratories, Inc. is now Director of Commercial Development for Oxford Paper Co., New York, N. Y.

**Richard J. Jacobus,** formerly of Wausau Paper Mills is now with Monsanto Chemical Co., St. Louis, Mo.

**James D. Johnson,** formerly of the Continental Can Co. is now Manager of Doubleday, Inc., Smithsburg, Md.

**Ralph C. Keef,** formerly of Alaska Lumber & Pulp Co. is now with St. Croix Paper Co., Woodland, Me.

**Khrishna K. Khandelwal,** formerly of Beloit Iron Works is now Director of Technical Services for Overly's, Inc., Neenah, Wis.

**Rudolf F. Kozich,** formerly of Dorr-Oliver, Inc. is now a project engineer for E. D. Jones Corp., Pittsfield, Mass.

**John H. Langstaff,** formerly of J. C. Wilson, Ltd. is now Sales Manager of Formex Co. of Canada, Montreal, Que.

**William H. Lehmkuhl,** consultant, has transferred his office from New Orleans, La. to 3120 Pleasant St., Hamilton, Ohio.

**Bengt Leopold,** formerly of The Mead Corp. is now Director of ESPRA and Professor of Pulp and Paper Technology at New York State College of Forestry, Syracuse, N. Y.

**John O. Logan,** Executive Vice-President of Olin-Mathieson Chemical Corp. has transferred his office from Baltimore, Md. to New York, N. Y.

**Solomon Marcovitch,** formerly of S. A. L'Air Liquid is now Manager of Black-Clawson France, Paris, France.

**J. Roger Marty,** formerly of Millspaugh, Ltd. is now Manager of Societe B. M. B., Paris, France.

**John J. McDonald,** General Manager of the Pulp Floc Division of the Brown Co. has moved his office from Boston, Mass. to 733 Third Ave., New York, N. Y.

**Joe B. Morrison,** formerly of Weyerhaeuser Co. is now Project Engineer with Southwest Forest Industries, Inc., Snowflake, Ariz.

**Orville D. Mussey,** formerly with the U. S. Geological Survey is now with Harza Engineering Co., Chicago, Ill.

**William H. Neuss** of the Borden Chemical Co. has been transferred from Bainbridge, N. Y. to Peabody, Mass.

**Clarence S. Nichols, Jr.** of the International Paper Co. has been transferred from Chisholm, Me. to Glens Falls, N. Y. as Assistant Division Engineer.

**Dudley J. Nixon** is now Assistant Director of the Paper Department of Geigy Dyestuffs Div., New York, N. Y.

**Yas Nomiyama,** research coating chemist for St. Regis Paper Co. has been transferred from Deferiet, N. Y. to W. Nyack, N. Y.

**John F. O'Flaherty,** formerly of Alliance Paper Mills, Ltd. is now Coating Superintendent for Bathurst Power & Paper Co., Bathurst, N. B.

**James D. Parmele** of St. Regis Paper Co. has been transferred from Deferiet, N. Y. to Jacksonville, Fla. as Administrative Assistant to the Vice-President.

**Richard W. Phelps** is now Vice-President of the Black-Clawson Co., Fulton, N. Y.

**Charles J. Prawdzik** of St. Regis Paper Co. has been transferred from Cleveland, Ohio to Pittsburgh, Pa. as Assistant General Production Manager.

**Robert W. Reed** of St. Regis Paper Co. has been transferred from New York, N. Y. to Deferiet, N. Y. as Director of Quality Control.

**John Schornagel,** formerly of H. P. Smith Paper Co. is now Director of Research and Development of the Pacific Coast Foil Co., So. San Francisco, Calif.

**George F. Schunning** is now General Sales Manager of J. O. Ross Engineering Division, Midland Ross Corp.

**John L. Sigler** is now Vice-President of Morden Machines Co., Portland, Ore.

**Igor Sobolev,** formerly of Rayonier, Inc. is now in the Organic Chemicals and Applications Department of Shell Development Co., Emeryville, Calif.

**Robert O. Stephenson** is now Division Manager of Champion Papers, Inc., Hamilton, Ohio.

**Lionel M. Sutherland,** formerly of Sutherland Refiner Corp. is now President of Tomlon Corp., Trenton, N. J.

**W. Donald Thompson** of Hercules Powder Co. has been transferred from Wilmington, Del. to Covington, Va. as Manager of Research and Development of the Fiber Development Department.

**Patrick C. Trotter,** formerly student at The Institute of Paper Chemistry is now a research associate with Weyerhaeuser Co., Longview, Wash.

**Kelevi A. E. Vesterinen,** formerly of Jujo Paper Co. is now Superintendent of Tampella Oy, Tampere, Finland.

**Leslie L. Warner,** formerly of Owens Corning Fiberglas Corp. is now a chemist with Galbar, Inc., Carlsbad, N. M.

**Robert C. Whitney,** formerly of Oxford Paper Co. is now with Spencer Chemical Co., Merriam, Kan.

**Jack E. Wilber** of St. Regis Paper Co. has been transferred from Deferiet, N. Y. to W. Nyack, N. Y. as Manager of Development at the Technical Center.

**Roger S. Williams,** formerly of R. T. Vanderbilt Co. is now a chemist with Southwest Forest Industries, Inc., Snowflake, Ariz.

**William H. Wiseman,** formerly of West Virginia Pulp & Paper Co. is now with Brunswick Pulp & Paper Co., Brunswick, Ga.

**Czeslaw J. Witkowski,** formerly of Dorr-Oliver Co. is now a staff engineer with Kimberly-Clark Corp., Neenah, Wis.

**Hirotsugu Yasuda,** formerly student at the New York State College of Forestry is now a research chemist at the Camille Dreyfus Laboratory, Research Triangle Institute, Durham, N. C.

* * *

**B. P. Knol** has succeeded **F. M. Muller** as the corporate representative of Netherlands Experimental Station for the Utilization of Straw, Groningen, Netherlands in the Technical Association.

**W. A. Liebenberg** has succeeded **G. J. J. Haumann** as the corporate representative of Masonite (Africa), Ltd., Estcourt, Natal, So. Africa in TAPPI.

**Hjalmar Madelung,** Technical Director, has succeeded **Paul Schou** as the corporate representative of the United Paper Mills, Ltd., Copenhagen, Denmark in the Technical Association.

**S. H. Watkins,** Assistant Sales Manager of the Pine and Paper Chemicals Department has succeeded **W. D. Thompson** as the official representative of Hercules Powder Co., Wilmington, Del. in TAPPI.

# EMPLOYMENT SERVICE

The rates per issue for Positions Open advertisements in *Tappi* are as follows:

| | |
|---|---|
| Per line | $ 3 |
| 1/16-page Box | $ 50 |
| 1/8-page Box | $ 80 |
| 1/4-page Box | $125 |

POSITIONS OPEN

P2055-61. Assistant managership, prominent eastern company producing pulp and coated paper. Good education and experience required. Resumés confidential.


## Physical or Organic Chemist

Creative physical or organic chemist with M.S. or Ph.D. or equivalent in experience wanted for research on products utilizing petroleum wax. Recent polymer developments have opened doors for research on novel wax applications. Experience in wax or polymer coatings, polymer applications, or packaging research beneficial but not essential.

This position is with the expanding Research Department of a major oil company in a friendly southwestern city of 25,000 Applicants with initiative and drive will find challenging opportunities for professional advancement. Excellent research facilities. Comprehensive employee benefits.

Confidential consideration: Send complete résumé of education, experience, and salary requirements to: **Personnel Relations Department No. 4, Continental Oil Company, Ponca City, Oklahoma.** **(P2047-61)**

## OPPORTUNITY FOR TECHNICAL SERVICE SPECIALIST

Chemical Engineer or Chemist preferred. At least 2 years direct paper mill experience. Must have interest in practical solutions to operating problems. Work in direct support of Sales Department. Approx. 25% travel.

Submit resume including education, experience and salary requirements to:

Manager of Selection & Placement

**GENERAL MILLS, INC.**
**9200 Wayzata Blvd.**
**Minneapolis 26, Minnesota**

(P2056-61)

## PAPER MILL ENGINEER

Graduate mechanical engineer wanted by leading manufacturer of fine business, printing and technical specialty papers. Require man experienced in estimating and layout of paper machines and auxiliary equipment.

This growing, multi-mill organization has its headquarters in a small community in Central Wisconsin, in the heart of outstanding recreational facilities.

Please send detailed resume of education and experience to:

**Employment Director**
**Nekoosa-Edwards Paper Company**
**Port Edwards, Wisconsin** **(P2057-61)**

## MANAGER PAPER RESEARCH

The expansion of our research and development activities has created an opening for a man with background in paper technology to manage the research and applied development work on specialty industrial papers. Work will involve the use of natural and synthetic paper making fibres in combination with polymer materials.

Candidate should preferably hold an advanced degree and possess a practical background in paper manufacturing. Previous supervisory experience desirable.

Our new research laboratory is ideally located in a university town offering excellent housing and schools near large metropolitan cities and famous recreational facilities.

Apply in confidence detailing education, experience, and salary desired to M-66, P. O. Box 2066, Phila. 3, Pa.

(P2059-61)

## NCR CHEMICAL RESEARCH CHEMICAL DEVELOPMENT

NCR'S Chemical Development Laboratory is engaged in a comprehensive study of record keeping and reproduction processes. An exceptional career opportunity exists in this challenging field for a qualified MS or PhD chemist. The man we are looking for should have a strong background in Organic or Physical Chemistry with 2–7 years' experience in paper coating formulation and development work. Primary duties will involve the development of new functional coated paper products with the opportunity of carrying ideas from the laboratory through pilot plant scaleup and ultimate commercial production. To be considered for this or other positions at our modern, R & D Center in Dayton, Ohio, write to: **T. F. Wade, Technical Placement, The National Cash Register Company, Dayton 9, Ohio. AN EQUAL OPPORTUNITY EMPLOYER**

**(P2060–61)**

## PROJECT ENGINEER— PULP AND PAPER DIVISION

We need a Chemical or Mechanical Engineer with 10–15 years of responsible experience and demonstrated ability as a Project Engineer on new major engineering or expansion programs of pulp and paper mills.

Opportunity for advancement good. Salary open. Location is Cleveland, Ohio. Please send complete resume and salary requirements to: **J. T. Wolcott, Personnel Manager, The H. K. Ferguson Company, 1783 East 11th Street, Cleveland 14, Ohio**

**(P2061-61)**

## PHYSICAL CHEMIST

Ph.D. or equivalent required with advanced training and interest in the chemistry and physics of surfaces. Prefer a man skilled in optical techniques to build understanding of the forming of polymer systems into barrier layers.

## PHYSICIST OR PHYSICAL CHEMIST

Require a Ph.D. or equivalent with an interest in rheological behavior of fiber systems to lead studies of treatment and behavior of pulp and other fibers in suspension.

These positions are available as a result of the recognition by a major producer of pulp, paper and packaging materials webs of the need for fundamental understanding to guide, improve and generate production. They will be part of a growing effort to be based on technical excellence. New laboratory facilities are being created in a rural location adjacent to several major metropolitan centers.

Please address all replies in absolute confidence to P2052-61, *Tappi*, 360 Lexington Ave., New York 17, N. Y.

## DIRECTOR OF PAPER CHEMICALS RESEARCH

A fine growth opportunity is being created by a large Chicago headquartered company.

This position requires an experienced paper mill man, thoroughly trained and experienced in paper mill operations. His specialty should be coated printing papers. A PhD or Masters degree in Chemistry or Chemical Engineering is highly desirable.

Man selected will organize and staff a research and development group to evaluate present research data and company products . . . then follow through with necessary efforts to bring products to marketing stage. Must be able to provide technical assistance to the sales department in latter stages of development.

This man is probably now a Technical Director or Assistant, earning approximately $12,000 per year, and looking for a real chance to show his ability. Salary is open.

If you would like to know more, please write in confidence to: **P2062-61, *Tappi*, 360 Lexington Ave., New York 17, N. Y.**

## DIRECTOR OF RESEARCH AND DEVELOPMENT

This is a Senior Management position requiring sound fundamental knowledge of cellulose chemistry. Experience in the dissolving wood pulp field is desirable.

The successful applicant should hold a Ph.D and have the ability to organize and direct a research and development group of some 18 professional and 17 supporting personnel. He will report directly to the Senior Executive Officer of the Company.

This challenging opportunity offers great scope in a growing, progressive organization operating a dissolving sulphite pulp mill at Prince Rupert, B.C., and a new bleached kraft pulp mill at Castlegar, B.C.

*Please write in confidence to:*

COLUMBIA CELLULOSE COMPANY, LIMITED
*1030 West Georgia Street, Vancouver 5, B.C.*

ATTENTION: EXECUTIVE VICE-PRESIDENT

V8656-1

# INDUSTRY NOTES

## *Paper Manufacturers and Converters*

APPLETON COATED PAPER CO.

The Appleton Coated Paper Co. announced the promotion of three laboratory employees. Two of the positions are newly created and the third has been in operation since May of this year. **Thomas W. Busch** was named to the position of assistant technical director, **William H. Page** was promoted to section head of Product Development, and **Roland F. Meyer** was named as section head of quality control.

BROWN CO.

**Walter N. Kinsley,** of Rowayton, Conn., has been named sales manager of Brown Co.'s newly formed Printing Papers Division, according to Edward H. Petrick, vice-president and general manager of the New Hampshire firm's Paper Division.

CHAMPION PAPERS, INC.

Leo Geiser, vice-president, Manufacturing, Champion Papers, announced that **Robert O. Stephenson** will succeed **Earl C. Jones** as division manager of the Ohio Division in

*Robert O. Stephenson, Champion Papers, Inc.*

*Earl C. Jones, Champion Papers, Inc.*

response to Mr. Jones' request to be relieved of the "heavy responsibilities of the division manager's office." At the request of Karl R. Bendetsen, Champion president, Mr. Jones agreed to continue to serve the division in a consulting capacity.

CONTINENTAL CAN CO.

**H. Ahlmeyer** has been named manager of production engineering for the Folding Carton & Drum Division, Continental Can Co. Succeeding Mr. Ahlmeyer in his previous position as manager of the Piermont, N. Y. folding carton plant is **G. Posson,** previously assistant manager in this plant.

CONTINENTAL CAN CO. OF CANADA LTD.

**A. G. Schumaker,** manager of the Toronto mill of Continental Can Co. of Canada Ltd., has been appointed manager of production engineering—mill operations.

Mr. Schumaker is succeeded as manager of the Toronto mill by **Charles E. Whitten,** plant manufacturing engineer at the company's Dominion mill. Mr. Whitten joined the company in the engineering department at the Dominion mill in 1951.

GREAT NORTHERN PAPER CO.

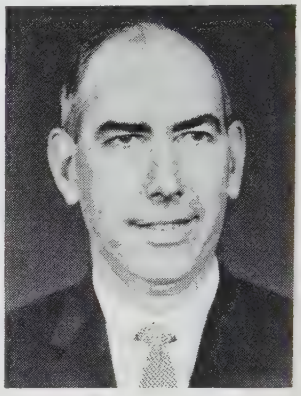
M. C. McDonald, president of Great Northern Paper Co., has announced the company's decision to enter the coated paper field in 1962, as part of its diversification and new product program.

At its recent meeting, Mr. McDonald disclosed, the Board of Directors authorized the new program at an estimated investment of $5,000,000 to $6,000,000 for equipment required to manufacture coated papers at the company's Millinocket mill. This investment will cover the cost of a new building, a coater, and related equipment. The base stock for coating will come from presently unused paper manufacturing capacity at Millinocket.

Construction will begin at once, and it is expected that the new plant will be in production in the last quarter of 1962.

INTERNATIONAL PAPER CO.

**C. S. Nichols** has been named assistant division engineer for International Paper Co.'s Northern Division. Mr. Nichols will be located in Glens Falls, N. Y., headquarters for the Northern Division, which includes paper mills at Chisholm, Me.; Ticonderoga, North Tonawanda, Corinth, and Niagara Falls, N. Y.; and York Haven, Pa.



*C. S. Nichols, International Paper Co.*

MENASHA WOODEN WARE CORP.

*Triangle Container Corp.*

Menasha Wooden Ware Corp., of Menasha, Wis., has agreed to purchase the assets of Triangle Container Corp., of Chicago, it was jointly announced today by R. L. Johnson, president of Menasha Wooden Ware Corp., and Norman Albin, president of Triangle Container Corp.

Triangle Container Corp., with its plant at 4343 W. Ohio St., founded in 1939, manufactures and sells corrugated containers in the Chicago area. Mr. Albin will continue to be in charge of the Triangle operation, and no change in Triangle's personnel is contemplated. These additional facilities will supplement Menasha's current sales and manufacturing activities in Chicago.

Menasha Wooden Ware, founded in 1849, is a diversified manufacturer of corrugated containers, pulp and paper, plywood, lumber, and wood flour. It has corrugated box plants at Menasha, Wis.; Anaheim, Calif.; Rockford, Ill., and an affiliated company at Winchonton, Mass.; pulp and paper mills at Otsego, Mich., and North Bend, Ore.; plywood plants and sawmill at North Bend, Ore., and wood flour plants at North Bend, Ore., and Tacoma, Wash.

OXFORD PAPER CO.

Announcement was made by William H. Chisholm, president of Oxford Paper Co., of the appointment of

**Robert T. Hart** as director of commercial development. In his new position with Oxford, Dr. Hart will report to Harold M. Annis, vice-president, Research and Development. He will continue for the present to reside in Portland, Me., but will be spending a large proportion of his time at the company's mills.

ST. REGIS PAPER CO.

*Service Award*

Nearly 4500 employees of St. Regis Paper Co.—representing over 55,000 years of service—will receive awards this year in recognition of five or more years of service with the company. The awards are scheduled for presentation at numerous ceremonies in the mills, plants, and offices of all divisions of St. Regis in the United States and abroad. In all, 4438 persons—or roughly one-seventh of the company's total number of employees—will be honored this year as part of St. Regis' continuing service awards program.

*Appointments*

St. Regis Paper Co. announces that **O. R. Titchenal** has been appointed manager of packaging machinery for the Engineering and Machine Division. Mr. Titchenal will continue to be located at the divisional headquarters in Providence, R. I. He will report to Donald O. Sensenbach, general manager of the Engineering and Machine Division.

**I. W. Miller** has been appointed director of engineering of the Engineering and Machine Division. Mr. Miller will continue to be located at Providence, R. I., and will report to D. O. Sensenbach, general manager of the Engineering and Machine Division.

SCOTT PAPER CO.

**Charles D. Dickey, Jr.,** a vice-president of Scott Paper Co., has been elected to the corporation's board of directors to fill the vacancy created by the resignation of Stanley Resor.

STRATHMORE PAPER CO.

The Strathmore Paper Co. has announced four changes affecting technical personnel.

Henry D. Johnston, president, announced that **James C. Gallatin, Jr.,** has been appointed technical director of the company, succeeding **Percival P. Godding,** who retired on August 31 after a 30-year career with the company. **Franklin G. Burr** has been appointed assistant technical director, and **Maurice E. Blew** has been appointed quality control manager.

SOUTHERN PINE LUMBER CO.

Announcement of the promotion of **Carl H. Shepherd, Jr.** to the position of production manager of Fiber Products Division has been made by Southern Pine Lumber Co., Diboll, Tex.

U. S. GYPSUM CO.

The United States Gypsum Co. has successfully started up its new paper mill in Jacksonville, Fla. Producing a range of paper grades for the company's entire line of gypsum board products, the new mill's major production equipment was supplied by The Black-Clawson Co.

A 9-cylinder paperboard machine trimming 156 in. and producing 150 tons every 24 hr. is the mill's main piece of equipment. It was built at the Watertown, N. Y., plant of the Paper Machine Division of The Black-Clawson Co. and is equipped with a suction return drum press, four primary presses, plain first main press, and a suction second main press. Black-Clawson's Autoflyte reel and Rollflyte Vertishear slitter-winder follow a double-decked drier section and twin calender stacks.

A 150-ft. long Transite hood and vapor absorption system was supplied by Black-Clawson's Air Systems Division in Hamilton, Ohio. The hood is equipped with a continuous aluminum plenum and a damper for adjusting the exhaust pick-up at any point in the hood. One of the hood's unusual features is a continuous walkway providing accessibility to rope carrier supports.

The three stock preparation systems which deliver all of the filler, back liner, and top liner stock for the paperboard machine were also built by the Black-Clawson Co. in the Middletown, Ohio, plant of the Shartle Division. Two Hydrapulpers, Liquid Cyclones, a Sutherland breaker trap, Brammer consistency regulators, Black-Clawson jordans and thickeners, Selectifier screens, and Black-Clawson pumps, agitators, and valves are included in the three systems.

S. D. WARREN CO.

S. D. Warren Co. has been selected for the second consecutive year for a first place award for having issued the best stockholder annual report of the pulp and paper industry in the 21st Annual Report Survey sponsored by *Financial World,* national weekly magazine. More than 5000 reports were reviewed in 96 categories during the 1961 competition.

WEST VIRGINIA PULP AND PAPER

Three executives at the Covington, Va., research laboratory of West Virginia Pulp and Paper Co. have been promoted to new responsibilities.

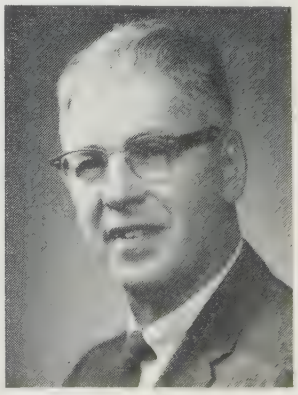
**Fred H. Freuler,** director of the Covington research laboratory since 1959, has been promoted to the newly-created position of director of papermaking and mechanical research and development. He will have overall responsibility for coordinating papermaking and mechanical research at Westvaco laboratories and will be headquartered in the company's New York offices.

**Girard L. Calehuff,** group leader of fluid mechanics re-

*James C. Gallatin, Jr., Strathmore Paper Co.*

*P. P. Gooding, Strathmore Paper Co.*

*Franklin G. Burr, Strathmore Paper Co.*



*Maurice E. Blew, Strathmore Paper Co.*

search at Covington, succeeds Mr. Freuler as research director. In his new assignment he will be responsible for research in the important areas of fluid mechanics, thermodynamics, applied mechanics, microbiology, development engineering, and converting.

**Joseph J. Kilian,** a research group leader attached to the Covington laboratory, has been promoted to research director responsible for papermaking process development work. Mr. Kilian's research groups provide papermaking process control services to operating divisions and provide technical assistance to the operating divisions in the startup of new papermaking equipment. Mr. Kilian's research groups have been attached to the company's Luke, Md., mill during recent months but are expected to return to Covington in early 1962.

WEYERHAEUSER CO.

Three new basic products have been successfully extracted from Douglas-fir bark by researchers at Weyerhaeuser Co.'s technical center in Longview, Wash.

W. H. Meadowcroft, manager of the company's new products division, announced introduction of WEF, Wey-Chem, and Firwax to potential market users. More than 10 years of research and development effort is behind the new products.

The new Wey-Chem, WEF, and Firwax product groups are chemically derived from bark and constitute a new development beyond Weyerhaeuser's Silvacon bark products, which have been produced by mechanical fractioning.

WEF (Weyerhaeuser Extracted Fibers) is manufactured by chemically dissolving away nonfibrous portions of the bark, leaving a pure, spindle-shaped bark fiber. It is a lignocellulosic fiber that has three major applications in thermosetting molding.

WEF replaces sisal as a reinforcing material in polyester premix compounds. Polyester premixes made of WEF replace phenolic molding compounds. And WEF may be used also as a reinforcing material in phenolic compounds.

Wey-Chem products are derived from a liquor which results from the primary extraction process. They are used as dispersants, binders, and adhesive ingredients.

Firwax, extracted from the liquor which results from the primary extraction, is arousing interest in two initial market applications: as an ingredient in investment casting waxes and as a hard wax in carbon paper inks.

## *Educational and Research Institutions*

U. S. FOREST PRODUCTS LABORATORY

"I wanted to devote more time to research." That's the reason Dr. Margaret Seikel gives for leaving the job as chairman of the chemistry department at Wellesley College to become one of the few women in the world who are studying lignin structure determination.

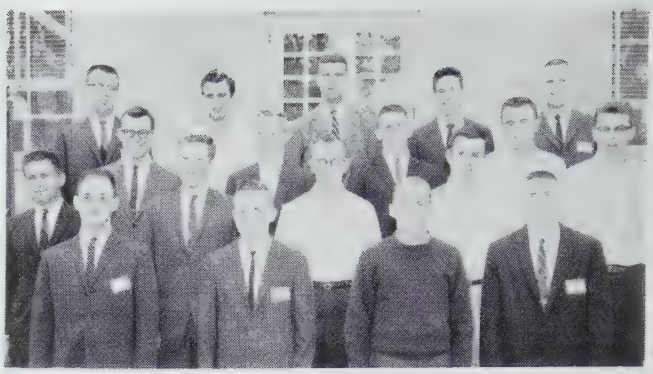
Dr. Seikel, who has taught college chemistry for 21 years, recently accepted a position as an organic chemist at the U. S. Forest Products Laboratory here, maintained by the Forest Service, U. S. Department of Agriculture. The Laboratory is currently conducting investigations to determine the basic structure, composition, and chemical characteristics of lignin, which is nature's cement for holding the cell walls of wood together. Lignin, which chemically makes up approximately one-third of wood, at present has no major industrial use. Dr. Seikel will be the associate of John C. Pew, who is the head of the lignin research at the Laboratory.

INSTITUTE OF PAPER CHEMISTRY

*Thirty-Second Class Enters Graduate School*

Seventeen students are enrolled in the 32nd class to enter the graduate school of The Institute of Paper Chemistry.

The new students represent bachelors degrees from 13 colleges and universities, and are from 14 different states.



***The 32nd class to enter the graduate school of The Institute of Paper Chemistry at Appleton, Wis.***

They are starting a graduate program which will take approximately four years to complete for the Ph.D. degree. The M.A. degree is normally awarded at the Institute after two years of graduate study.

Each new student, with all other regular students of the Institute who are United States citizens, is awarded a full time scholarship. Students devote full time to study. No work is done by students on any cooperative project carried on in the Institute's research laboratories, or on projects being pursued by Institute staff members. With total registration of students in the regular student body numbering 75, the Institute will this year award approximately $213,000 in scholarships.

*Faculty Appointment*

The Institute of Paper Chemistry has announced the appointment of **Martin C. Mathes** to its faculty in the biology department. Dr. Mathes was appointed to the Institute's staff in July as a research aide in the biology group. His work will be in the area of forest genetics.

LEHIGH UNIVERSITY

*Printability Symposium*

TAPPI Graphic Arts Committee members were among the 45 technical representatives of graphic arts industrial and research organizations who attended an invitational Printability Symposium at Lehigh University on Thursday, Oct. 26, 1961. The purpose of the conference, which was sponsored by the Lehigh printing research group, was to de-

***TAPPI Ink Receptivity Subcommittee confers with Lehigh research personnel prior to the Lehigh Printability Symposium. Seated William C. Walker, West Virginia Pulp and Paper Co.; Mrs. Ann Fisch, Lehigh; J. R. Gunning, Abitibi Power and Paper Co. Standing: Herbert J. Connell, S. D. Warren Co. and William D. Schaeffer, Lehigh***

***Miss Constance Grund, Lehigh, demonstrates IGT printability tester with grindometer disk and blade applicator to William C. Walker, who designed the modifications***

scribe recent results and trends in fundamental research on ink-paper-press relationships.

Included on the symposium agenda were reports from two TAPPI-sponsored projects—Surface Strength Evaluation (TAPPI Appropriation Nos. 130 and 166) and the Transfer and Penetration Aspects of Ink Receptivity (Nos. 168 and 182). Progress on the latter project was the major topic of discussion at a presymposium meeting of the Ink Receptivity Subcommittee with Lehigh research personnel on Wednesday, October 25. TAPPI was represented at the committee meeting by the Ink Receptivity Subcommittee chairman, William C. Walker, West Virginia Pulp and Paper Co.; Graphic Arts Committee Chairman Herbert J. Connell, S. D. Warren Co.; and A. P. Reynolds, S. D. Warren Co. Other members of the subcommittee are Joseph Curado, Sun Chemical Corp., and Maxwell Hughson, Consolidated Water Power and Paper Co.

The symposium, which was held in Packer Hall, the Lehigh University Center, was chairmanned by A. C. Zettlemoyer, distinguished professor of chemistry and research director of the National Printing Ink Research Institute. Registrants also toured the graphic arts and related surface chemistry research laboratories in the William H. Chandler Chemistry Building. The conference and tours were organized by Miss Jacqueline Fetsko, NPIRI assistant research director.

In welcoming the group, Harvey A. Neville, University president, cited the importance of industry-university cooperation in the pursuit of fundamental research. He also emphasized benefits to graduate students who are given the opportunity for advanced study. Dr. Zettlemoyer then outlined the master plan of the Lehigh research program which is designed "to uncover basic factors contributing to printing results."

Following a review of research techniques by Samuel I. Cossens, supervisor of Printing Research at Lehigh, Miss Fetsko discussed results of a comparison of four laboratory printmakers developed in Europe. Her findings were that cylinder-type presses transferred less ink than flat-bed presses and also responded more to changes in printing speed.

William D. Schaeffer, associate director of NPIRI, discussed the effects of printing conditions on ink transfer and print quality. The systematic study, which involved two years of work under the TAPPI Ink Receptivity grant, showed that different inks and papers respond differently to changes in printing conditions. A differential equation was derived to predict trends.

The afternoon session was opened with a talk by Gosta Carlsson, director of the Swedish Graphic Arts Research Laboratory in Stockholm. Mr. Carlsson, who is currently touring research centers in the United States, described his recently developed GFL rotary press designed specifically for newsprint testing.

Joseph J. Hammel, Sun Chemical Corp. fellow in chemistry at Lehigh, related his efforts to measure particle size and distribution of pigments in their final dried state by light scattering techniques. The need for such measurements resulted from his previous study of colored prints made from phthalocyanine blue pigments.

Surface strength evaluation of paper, also carried out under a two-year TAPPI grant, was discussed by Miss Fetsko. The speaker outlined the procedures recommended by the Lehigh study for performing pick tests on an accelerating speed type of printmaker. She also demonstrated how picking variables can be applied to fundamental concepts of splitting forces as suggested by Stefan's equation.

Discussion of future work to be undertaken concluded the conference. Dr. Zettlemoyer told the group that, while the study of the printing process *per se* must be continued, emphasis must now be placed on idealized experiments to explain events before, at, and immediately after the moment of impression.

Highlight of the laboratory tour was the initial showing in the United States of the FOGRA printability tester, a German-developed miniature test press. Another focal point was the IGT printability tester developed in Holland but modified in the United States with a grindometer disk and blade applicator. The modification demonstrated was the design of Dr. Walker while he was active on the TAPPI Surface Strength Subcommittee.

Assisting with the tour, which featured research printing equipment, rheological instruments, paper and print measuring devices, and surface chemistry experimentation, were Robert Schiesser, supervisor of Dispersion Research; Anthony Butto and Nicholas Halkius, graduate assistants; Harold J. Stanley, Jr., Lewis Calder Foundation fellow; and Miss Constance Grund, Mrs. Ann Fisch, and Mrs. Jean Buckingham, research assistants.

***Miss Jacqueline Fetsko showing the Fogra printability tester, a German developed instrument, to Gosta Carlsson, Swedish Graphic Arts research director, and A. C. Zettlemoyer, NPIRI research director***

*Graphic Arts Fellows*

Recipients of five Fellowship Awards for advanced study in graphic arts and related fields for the 1961–62 academic year have been announced by Robert D. Stout, dean of Lehigh University's Graduate School.

Winners of three fellowships granted to Lehigh for fundamental research relating to printing ink are **Joseph J. Hammel,** awarded The Sun Chemical Corporation Fellowship; **David R. Bassett,** the Howard Flint Fellowship, and **Harold D. Stanley, Jr.,** Louis Calder Foundation Fellowship.

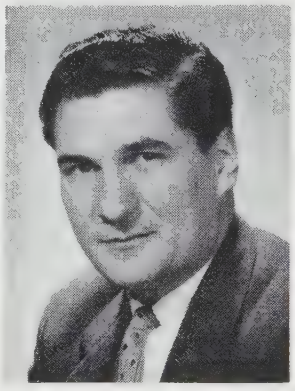
Graduate assistantships for printing ink research have also been awarded to **Nicholas Halkias** and **Anthony Butto,** both graduates of Pennsylvania State University.

*Appointment to Committee*

**Albert C. Zettlemoyer,** distinguished professor of chemistry at Lehigh University, has been named to a three-year term as chairman of the Committee on Colloid and Surface Chemistry, in the National Academy of Sciences-National Research Council.

McGill University

**Andrew J. Bobkowicz** has been named as the recipient of the 1961–62 D. S. and R. H. Gottesman Foundation Scholarship at the Pulp and Paper Research Institute of Canada and McGill University for the third successive year. The Gottesman Foundation has awarded this scholarship each year since 1942 for the purpose of supporting postgraduate research of interest to the pulp and paper industry, and Mr. Bobkowicz will use the funds to continue his final year of study and research for the Ph.D. degree at the Institute.



*A. J. Bobkowicz*

Under the supervision of W. H. Gauvin of the Institute's Chemical Engineering Department, he is investigating the turbulent flow characteristics of model fiber suspensions. His work forms part of a larger project on the general subject of fiber behavior in flowing suspensions now being carried on at PPRI.

North Carolina State College

**Eric Louis Ellwood** has joined the faculty of the School of Forestry. Dr. Ellwood will be professor and head of the department of wood products—he will be responsible for the School of Forestry's undergraduate curricula in pulp and paper and wood technology and all graduate programs in wood products.

Dr. Ellwood is a native of Australia where he received his undergraduate degrees. His Ph.D. was earned at Yale University. He comes to State College from the University of California Forest Products Laboratory where he has been wood technologist and lecturer since 1957.

Purdue University

*Short Course Offerings in Packaging, 1961–62*

Industrial Packaging Technology, Purdue University, Technical Extension, Lafayette, Ind., Home Study Program, thirty lessons—Fee $50. The course covers, primarily, the field of packaging done in industrial plants and only incidentally that of consumer packaging. The thirty lessons included in the course deal with the following topics, among others: materials of packaging, shipping containers, warehousing, carloading, materials handling, military packaging, quality control, and testing.

Materials of Packaging, Purdue University, Lafayette, Ind., Home Study Course, thirty lessons—Fee $60. The course will cover all of the materials commonly used in packaging; a description of them, the methods used in their manufacture, their physical properties, and means for testing them and their uses. The thirty lessons included in the course, among others, deal with the following topics: (1) place of packaging materials in the U. S. economy, (2) history, development, and general theory of materials, (3) wood, veneer, plywood, etc., (4) pulp, paper, and paperboards, (5) steel, nails, wires, straps, and tinplate, (6) aluminum and other nonferrous metals, (7) films, plastics, adhesives, waxes, and fillers, (8) coatings, linings, and chemical treatments, (9) containers, cushioning materials, closure materials and glass, and (10) combinations of materials, specifications, testing, and quality control.

University of Maine

John Heuer, Bangor, vice-president of the Great Northern Paper Co., has been appointed by Frederic Soderberg, president of the University of Maine Pulp and Paper Foundation, to serve as chairman of the committee to arrange for the third Summer Institute for the pulp and paper industry at the University of Maine.

Mr. Heuer has served as a member of the Summer Institute Committee for the past two years during which Soderberg, then vice president of the Foundation, was chairman.

Others appointed to serve on the committee are Harold Annis, New York City, vice-president of research and development of Oxford Paper Co. and president of TAPPI; William Caddoo, New York City, vice-president of Continental Can Co., and a director of the University of Maine Pulp and Paper Foundation.

Prof. Lyle C. Jenness, head of the department of chemical engineering at the university, and director of the two institutes, and William Ketchen, Edmundston, N. B., vice-president-technical services of Fraser Companies, Ltd. will continue as members of the committees.

Thomas Mangan, Livermore Falls, a charter member of the Pulp and Paper Foundation and long active in many phases of the foundation program, will also serve. He is a retired mill manager for the International Paper Co. George Rand, New York City, vice-president of International Paper Co., will be the seventh member of the institute committee.

The summer institutes are sponsored jointly by the University of Maine Pulp and Paper Foundation and the University of Maine. They were established in response to a request from and as a service to the pulp and paper and allied industries. During each of the last two summers two three-week programs have been offered—one in pulp technology, and the other in paper technology. The committee will determine the dates for the institute and the program to be offered.

The Royal Institute of Technology (Sweden)

Announcement has been made that the King of Sweden, Gustaf Adolf VI, upon recommendation of the board of trustees of The Royal Institute of Technology, Stockholm (Sweden), and by action of the Swedish Cabinet, has appointed **Bengt G. Ranby** to two newly-created positions—professor of polymer technology and chairman of the Department of Polymer Technology, at The Royal Institute of Technology, Stockholm, Sweden.

Syracuse

The State University College of Forestry at Syracuse University announced recently that **Bengt Leopold** has been appointed director of the Empire State Paper Re-

search Institute and professor of pulp and paper technology, effective Oct. 1, 1961.

Dr. Leopold will direct the Empire State Paper Research Institute (ESPIRI), a research organization consisting of a team of 10 researchers jointly sponsored by the College of Forestry and the Empire State Paper Research Associates, Inc. (ESPRA). ESPRA is a nonprofit organization consisting of leading pulp and paper companies in the United States, Canada, Australia, and seven different European countries which has continuously sponsored research at the Syracuse Forestry College since 1945.

The research program is concerned with fundamental problems only—such as those pertaining to fiber structure and composition and its relationship to paper properties, chemical mechanisms involved in both oxidative and reductive bleaching, the energies involved in the mechanical separation of fibers, the nature of colored bodies in fibers and wood, and the qualities of high-yield pulps, particularly from hardwoods.

The State University College of Forestry at Syracuse University has also announced that **Robert H. Marchessault** has been appointed associate professor of forest chemistry and a member of the research staff of the college's Cellulose Research Institute.

### University of Wisconsin

*Industrial Adhesives Applications Institute*

The University of Wisconsin Extension Division in cooperation with the College of Engineering is conducting an Engineering Institute on Industrial Adhesive Applications, Jan. 16–17, 1962, in Madison. The fee for the two-day institute will be $25.

*Institute Objectives and Scope*

Occasionally it is necessary to take stock of some of the basic principles and fundamentals involved in adhesive usage. The solution to a troublesome problem can be traced too often to a mere violation of these principles. The main objective of this institute is to provide this review.

Registrants are asked to select a group, when they preregister, for the purpose of presenting their individual problems and for participating in group discussion and solution of fellow conferees' problems. The problems, discussions, and conclusions from each group will be recapped.

During the final general session, questions may be directed to the group leaders for clarification. The group leaders and speakers have been selected on the basis of the knowledge and experience they have in their respective fields. Design engineers, product stylists, value analysts and industrial engineers from companies using or considering using adhesives will not want to miss this informative program. Write to William W. Wuerger, Institute Coordinator, Engineering Department—Extension Division, University of Wisconsin, Madison, Wis., for additional information.

## *Industry Suppliers*

### Albany Felt Co.

Top production men from paper mills in Canada and across the nation were guests at Albany Felt Co.'s recent bleached kraft round table.

The three-day session was held at Albany Felt's, Albany, N. Y., plant, with Larry M. Woodside, chief engineer, Technical Field Services, moderating. Among the major topics under discussion were: shadow marking, finish, Durasorb needled felts, coating and felt cleaning and conditioning.

Representatives were present from mills in Ontario and Quebec, Canada, Washington, California, Virginia, Idaho, Florida, Minnesota, Alabama, North Carolina, Wisconsin, and Michigan.

### Louis Allis Co.

The Louis Allis Co., 437 E. Stewart St., Milwaukee, Wis., recently released Bulletin No. 112 that describes the operation of Syncro-Range Select-A-Spede drives. Ranging in size from 5 to 200 hp., these drives are suited for operation on multiunit conveyors, textile, or paper printing, etc.

### Allied Chemical Corp.

The technical service laboratory of Allied Chemical's General Chemical Division has been moved to new quarters at the company's Morris Township, New Jersey, research center. The new technical service facilities occupy part of the recently completed laboratory wing. Previous site was in Edgewater, N. J.

General's technical service laboratory includes departments concerned with application of the division's many industrial and specialty chemicals in such industries as refrigeration, aerosol, urethane foam, paper, detergent, fertilizer, water and sewage treatment and electroplating.

The new address is: Technical Service Laboratory General Chemical Div., Allied Chemical Corp., P. O. Box 405, Morristown, N. J.

### American SF Products, Inc.

Hinken and Co., Inc., of Pittsburgh, Pa., has been appointed to represent American SF Products, Inc., manufacturers of air handling equipment for the paper industry. Under the presidency of William Hinkle, Jr., this firm will handle SF equipment throughout Ohio, West Virginia, western Pennsylvania, and the two northern Maryland counties.

### Babcock & Wilcox Co.

*Boiler Division*

**Joseph Delano Hitch, Jr.,** has been appointed general manager, International Sales of The Babcock & Wilcox Co.'s Boiler division, it was announced here recently by L. S. Wilcoxson, vice-president. Mr. Hitch is former president and chairman of the board of Dorr-Oliver, Inc., where he had more than 33 years' experience in international operations. He will direct all international sales activities for B&W's Boiler division and will make his headquarters in New York.

*Marketing Department*

In line with a concerted effort to expand its share of world power equipment markets, The Babcock & Wilcox Co. has established a marketing department within its Boiler division, with **W. H. Rowand,** vice-president, in charge. Succeeding Mr. Rowand as head of the engineering department is **G. W. Kessler,** chief engineer since 1955.

Named to posts in the new marketing department are **F. E. Hutton,** as marketing manager, and, reporting to him,

*W. H. Rowand, The Babcock & Wilcox Co.*

*G. W. Kessler, The Babcock & Wilcox Co.*

**C. L. Marquez,** as manager of product planning and market research, **W. D. Stevens,** as proposition manager, and **D. R. Wilson,** as commercial manager.

*Houston Sales Office*

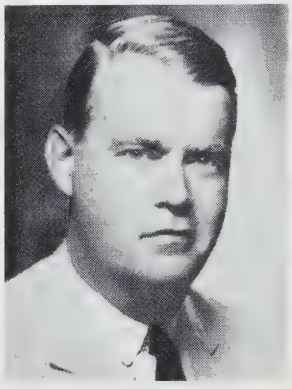
The Babcock & Wilcox Co.'s Tubular Products division has announced the relocation of their Houston Sales Offices, effective August 31. The new mailing address will be P. O. Box 25127, Houston 6, Texas.

Bird Machine Co.

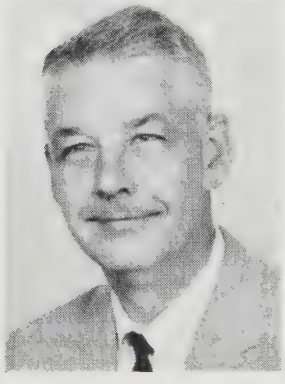
Bird Machine Co., South Walpole, Mass., has just issued an 8-page profusely illustrated bulletin on their stainless steel, free vortex, drop-in-pressure Cyclean units for removal of dirt and shines from pulp and paper stocks. Information is given on the range of sizes and applications. Described also is the new baffle head construction that prevents ingress of air in the larger sizes, and the sintered ceramic cones and tips that resist wear and erosion.

Black-Clawson Co.

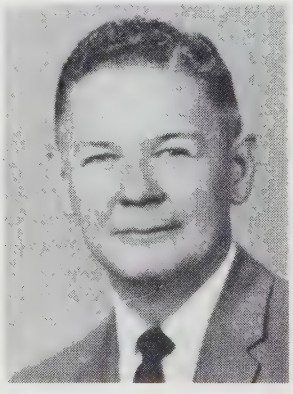
Frank T. Peterson, president, The Black-Clawson Co., announces that effective September 1, **Bruce Otis,** as assistant to the president, was made responsible for the administrative functioning of the company's international operations. Mr. Otis will be responsible for administrative coordination of all activities between the American divisions of the company and its manufacturing operations, sales offices, representatives, and licensees outside of the United States. He will maintain headquarters in the Corporate Office, New York, N. Y.



***Bruce Otis, The Black-Clawson Co.***



***Edward J. Kuhn, The Black-Clawson Co.***



***R. W. Phelps, The Black-Clawson Co.***

The Black-Clawson also announced the appointment of **Edward J. Kuhn** as a sales engineer in the company's Air Systems Division in Hamilton, Ohio, effective Oct. 9. 1961.

The appointment of **Richard W. Phelps** as vice-president of The Black-Clawson Co. has been announced. Mr. Phelps will continue his responsibilities as general manager of the Dilts Division, Fulton, N. Y., manufacturer of the Black-Clawson line of paper and paperboard coating and converting equipment and plastic extrusion machinery.

John W. Bolton & Sons, Inc.

*Bolton Emerson Southern, Inc.*

John W. Bolton & Sons, Inc., and The Emerson Manufacturing Co. Division, Lawrence, Mass., have purchased the Sheppard Plate and Machine Works of Atlanta, Ga., 41-year old manufacturers and nationwide distributors of disk refiner plates for the pulp and paper industry and attrition mill plates for the feed industry. As a result of this acquisition Bolton-Emerson Southern, Inc., has been formed as a new corporation affiliated with the parent company. Operation of the new company will be continued from the same location formerly occupied by Sheppard.

Borg-Warner Corp.

*Marbon Chemical Div.*

Marbon Chemical Division of Borg-Warner Corp. has named **Martin H. Schneider** technical sales representatives for a 21 central state area with headquarters in Chicago.

Bristol Co.

*Data-Logger Bulletin*

Data-Master® loggers perform two basic functions: (1) accurate, systematic collection of process, test and other operating data and (2) presentation of this data in the most useful form for supervisory, process analysis, research and accounting purposes or for direct input into computers.

A new bulletin (D 401) released by The Bristol Co., Waterbury 20, Conn., is written to alert potential users to the economy and, application flexibility of Data-Master automatic data loggers. Clear photographs supplement discussion of operating system installations in manufacturing, distribution, and process industries.

*Pneumatic Receiver*

A new Product Data (A113.3.3-2) describing the Series 670 Metagraphic® Pneumatic Receiver has also been released by The Bristol Co. Clear photographs and engineering drawings are used to supplement discussion of the instrument's construction, design, size, and capabilities.

The Series 670 is an important addition to The Bristol Co.'s line of "Functionally Unitized Instruments." Such instruments are engineered to be functionally compatible with a manufacturing or process system. Transmitting, controlling, and supervising instruments are placed—without grouping—in the most desirable location.

Cameron Machine Co.

Cameron Machine Co., Dover, N. J., in an extension of their sales coverage, has announced the appointment of **John Daly** and **Lewis Humberstone** to the Mid-West area. The new appointees will assume the coverage formerly done by R. Sherman who has resigned.

The resident service engineer in the Ohio area, **George House,** will continue in that capacity.

Chain Belt Co.

A new bulletin, No. 6175P, explaining features of its new, welded, all-steel, Uni-Barrel, conveyor chain has been announced by Chain Belt Co., Box 2022, Milwaukee, Wis. Designed especially for the pulp and paper and allied industries for rugged drag conveyor duty, Rex Uni-Barrel chain is ideally suited for conveying sawdust, chips, slags, pulp logs, and miscellaneous mill refuse.

Ciba Co., Inc.

A new yellow, Direct Bond Yellow P-A has been developed by CIBA Co., Inc. It is of particular interest to mills seeking a good economical greenish yellow as a base for greens, canary shades, or as a self color. Formulations based on Direct Bond Yellow P-A show substantial savings in many cases.

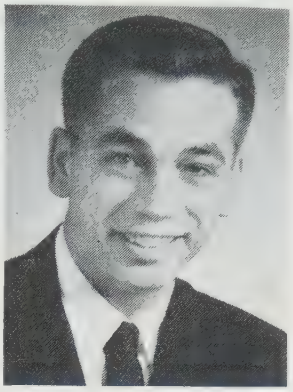
*Transfer*

**Preston Roy Harrington** has been transferred to the Los

Angeles office of CIBA Co., Inc. Mr. Harrington who will be in charge of the Los Angeles laboratory has been transferred from CIBA's Charlotte, N. C., district office. He has been with the company since 1956.

CORN PRODUCTS SALES CO.

**Daniel E. Lucas** has been appointed Eastern Regional Sales-Service manager for the Industrial Division, Corn Products Sales Co. Mr. Lucas replaces Clyde D. Woodburn, who was recently named manager of the Division's Philadelphia District office. Mr. Lucas will be located at the company's Eastern Region office, 10 E. 56th St., New York 22, N. Y.



*Daniel E. Lucas, Corn Products Sales Co.*

CRANE PACKING CO.

Crane Packing Co., 6400 W. Oakton St., Morton Grove, Ill., now offers a complete line of rubber expansion joints to industry. Made from a specially developed high-grade abrasion-resistant rubber that has been compounded for maximum flexibility and nonset characteristics, they are suitable for a wide range of applications. Standard spool type joints are available for pipe sizes from 2 in. up to 72 in. Vacuum type joints are designed for 30 in. of mercury and pressure type joints for 0 to 150 p.s.i., depending on size. Special joints can be designed in tapered, rectangle, oval or other shapes and for acids, gases, chemicals, etc.

DEZURIK CORP.

A new 8-page Bulletin, No. 302 is now available describing DeZurik rubber seated gate valves. The bulletin lists features of the valve as well as materials, dimensions, and actuators, and outlines the broad areas where the valve is extensively used. For a free copy, write to DeZurik Corp., Sartell, Minn., or DeZurik of Canada Ltd., Galt, Ont.

DIAMOND ALKALI CO.

Acquisition of Fiber Chemical Corp., Cliffwood, N. J., through an exchange of stock, has been announced by Raymond F. Evans, chairman and president, Diamond Alkali Co. A producer of organic chemicals and inorganic specialties, Fiber Chemical will be operated as a wholly owned subsidiary of Diamond Alkali Co. With annual sales of approximately one and one half million dollars, the firm is a supplier for the detergent, paper, textile and leather markets.

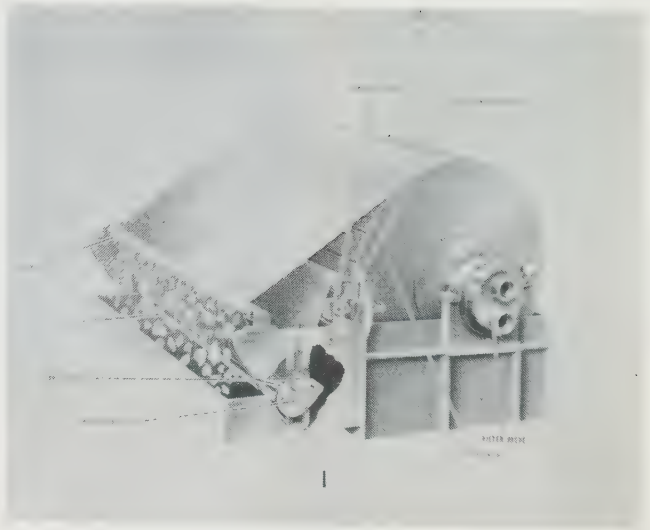
DORR-OLIVER, INC.

*Belt Filter*

A new belt filter design, which finally solves the related problems of smooth application of the medium to the filter drum and uniform and controlled belt tracking, has been introduced in a full range of sizes and materials by Dorr-Oliver, Inc., Stamford, Conn.

Known as the D-O Webtrol filter, the new unit applies dependable web tracking control and web spreading equipment to a new version of the basic Oliver vacuum drum filter design. A bowed roll spreads and smooths the fabric, holding the medium flat against the filter drum under uniform tension. A sensitive edge-position device operates through a modern feedback circuit to adjust continuously a tracking roll located so that it can position the belt directly on the filter drum.

The new D-O Webtrol filter, uses a three-roll system that provides positive belt control, continuous wrinkle removal,



*The D-O Webtrol filter—a new belt-type vacuum drum filter featuring positive belt control, continuous wrinkle removal, constant hydrostatic cloth backwash and friction-free belt travel*

constant hydrostatic cloth backwash, and a friction-free belt path. The filter medium passes over a bowed discharge roll, an adjustable roll, and a tracking roll in that sequence.

The bowed roll removes wrinkles by spreading the web, simultaneously providing initial separation of the cake for discharge.

It delivers a wrinkle-free belt to the adjustable washing roll. At this point in the web travel, both high velocity jet sprays and hydrostatic washing are used for thorough removal of surface solids.

DU PONT

**John A. Turner,** whose career with du Pont's Dyes and Chemicals Division spanned 42 years, has retired as manager of the Chicago district sales office. **Francis L. Shackelford, Jr.,** has been named to succeed him.

The retirement of **Robert R. Farwall** as manager of the New England district sales office of du Pont's Dyes and Chemicals Division and the appointment of **Harold L. Sager** to succeed him have also been announced by the company.

EASTMAN KODAK CO.

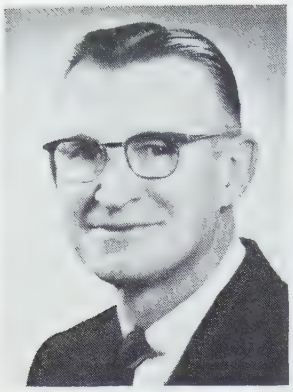
About 100 American colleges and universities have received a total of approximately $1 million in contributions under Eastman Kodak Co.'s 1961 aid-to-education program. The Kodak program calls for 58 direct grants, 44 fellowships awarded for advanced studies, and a variety of special contributions.

Direct grants totaling almost $300,000 were awarded to 58 privately endowed colleges and universities for 1961. The grants are based on the number of graduates from these institutions who joined Kodak five years ago and are presently employed by the company.

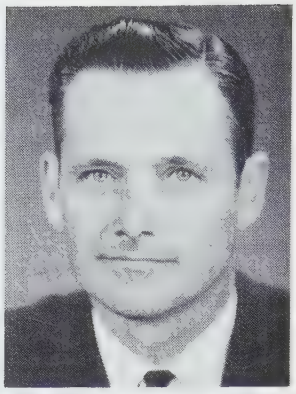
The grants help schools to compensate for the difference between the actual cost of educating graduates now with Kodak, and the amount that these individuals may have paid in tuition and fees. The grants also recognize the major role that the graduates are playing in the company's progress.

The 44 fellowships, previously announced and now in use, are valued at more than $150,000. Kodak awards the fellowships to colleges and universities who then select qualified individuals on the basis of financial need and ability.

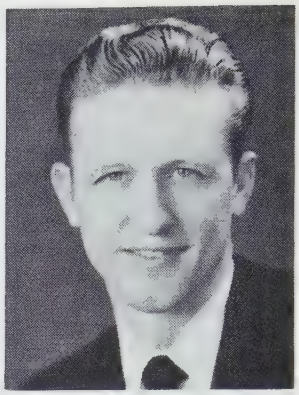
This year a total of 31 fellowships are being used by students working toward Ph.D. degrees—20 in chemistry, 3 in physics, 3 in chemical engineering, and 5 in business. The remaining



*Edward R. Huckman, The Foxboro Co.*



*Earle W. Pitt, The Foxboro Co.*



*Steve V. Kerstner, The Foxboro Co.*

*Dudley J. Nixon, Geigy Chemical Corp.*

13 fellowships were awarded to persons studying for M.S. degrees in engineering.

Fellowships awarded to students at eight Southern and three Southwestern universities were sponsored by Tennessee Eastman Co. and Texas Eastman Co., respectively. Both units are Kodak chemical divisions. The remaining 13 were sponsored by the parent company located in Rochester, N. Y.

The special contributions total more than $500,000 this year. They include special grants to educational institutions in areas where Kodak has major manufacturing interests and contributions to groups such as the United Negro College Fund, The National Fund for Medical Education, and the Empire State Foundation of Independent Liberal Arts Colleges.

Kodak has long recognized the important role of colleges and universities in preparing young Americans for mutually rewarding careers in industry. Founder George Eastman was a regular and often anonymous contributor to America's educational institutions, the fellowship program was begun in 1939, and in 1955 the company inaugurated a major new program of direct grants.

During the past five years Kodak's contributions under the aid-to-education program have amounted to approximately $4 million.

### Frank W. Egan & Co.

Edward F. Egan, president of Frank W. Egan & Co., has announced the appointment of **James A. Gibbons** of Richardson, Tex., as a sales engineer. Mr. Gibbons, who has been assigned to extrusion-coating equipment, represents the second addition within a month to the Somerville, N. J., firm's staff of sales engineers.

### Fiber Research Corp.

Fiber Research Corp. is pleased to announce that its directorship, a post left vacant since June by the death of Edgar G. Putnam, former director and founder, will be filled starting October 1 by **John R. Parkinson.** He will assume full responsibility immediately for the direction of the newly reorganized Fiber Research Laboratory which now offers complete testing, evaluation, and contract research and development facilities to the forest products industry. Located in Seattle, which is rapidly becoming the center for West Coast research activities in the fields of pulp and forest products, Fiber Research is the only independent commercial facility in the area fully staffed and equipped to undertake any investigations on behalf of its clients.

### Fischer & Porter Co.

*Stock Consistency Regulation from High Density Tower*

A new bulletin (90-26-15) published by Fischer & Porter Co., 4 Jacksonville Rd., Warminster, Pa., makers of process control instruments, describes three typical systems for consistency regulation from a high density tower. Two of the systems are for single-stage regulation, with dilution at one or two points; the final system involves two-stage regulation. The features of each system are outlined in the bulletin, and simple schematics are used to illustrate each. F&P instrument requirements are specified.

*Cascade Control*

Application Bulletin 91-53P-04, published by Fischer & Porter Co., 26 Jacksonville Rd., Warminster, Pa., outlines the elements of cascade control of chemical processes in fields such as paper, petroleum, chemical production, food processing, and metalworking.

### Foxboro Co., Ltd.

**Edward R. Huckman** has been named general sales manager for The Foxboro Co., Ltd. He will have charge of all industrial instrument sales activities for the Montreal firm and its branch offices in St. John, Toronto, Sudbury, Port Arthur, Edmonton, Calgary, and Vancouver.

**Earle W. Pitt** has been named Western field sales manager of The Foxboro Co., as regional manager of the Cleveland territory, and **Steve V. Kerstner** as branch manager of the company's Los Angeles office, replacing Mr. Pitt.

### Freeport Sulphur Co.

**Robert C. Hills** has been elected president of Freeport Sulphur Co., it was announced recently by Langbourne M. Williams, chairman of the board. He succeeds Charles A. Wight, who became vice-chairman of the board. Mr. Hills has been a director and executive vice-president of Freeport since 1955. He joined the company in 1934 as an assistant chemist at its Grande Ecaille sulfur mine in Louisiana. He was named assistant to the president in 1947, and elected a vice-president in 1950.

### Geigy Chemical Corp.

Geigy Dyestuffs, Division of Geigy Chemical Corp., Ardsley, N. Y., announces the appointment of **Dudley J. Nixon** to the position of assistant director, Paper Department. He will assist S. S. Harkavy, director, Paper Department, in directing dyestuff and chemical sales and technical service activities to the paper, pulp and converting industries in the United States and Canada. In addition to his new responsibilities, Mr. Nixon will continue to personally service the mid-Atlantic states assigned to him since 1951.

### General Aniline & Film Corp.

**John I. Snyder, Jr.,** of New York City was recently elected chairman of the Board and chief executive officer of General

Aniline & Film Corp. He succeeds Thomas A. Morgan who retired from the position as chairman.

The Board also elected **Ross D. Siragusa** of Chicago, Ill., as vice-chairman. Mr. Siragusa is president and chairman of the Board of Admiral Corp.

**Philip M. Dinkins,** president of General Aniline & Film Corp., will continue to serve in that capacity.

*Data Bulletin*

A comprehensive 40-page data bulletin has just been issued by the Commercial Development Dept., The Chemical Group, General Aniline & Film Corp., 435 Hudson St., New York, N. Y., on GANTREZ AN, the first in a series of new water-soluble copolymers. GANTREZ AN resin, which is available in several molecular weights, is a water-soluble polyelectrolyte. Chemically it is the interpolymer of methyl vinyl ether and maleic anhydride (PVM/MA).

GLIDDEN CO.

**John R. Dam** has joined the staff of The Glidden Company in the capacity of director, Paper Applications Laboratory. Mr. Dam has had extensive practical papermaking experience at Allied Paper Co., Kalamazoo, Mich. and Champion Paper and Fibre Co., Hamilton, Ohio. He was until recently the manager of Papermaking Research and Development at Champion Paper and Fibre Co., Hamilton, Ohio.

*Glidden Lectureship in Chemistry*

The Glidden Co. takes pleasure in announcing that The Institute of Paper Chemistry at Appleton, Wis., has been one of several universities and colleges chosen this year to be the recipient of an award which will permit them to present at Appleton a "Glidden Lectureship in Chemistry."

The speaker to be chosen, the subject of his lecture, and its time and place under Glidden policy is left entirely to the institution receiving the award. The Institute of Paper Chemistry will announce those details after they have concluded their arrangements.

GORHAM LABORATORIES, INC.

Gorham Laboratories, starting from a very modest beginning in a remodeled Maine farmhouse, has enjoyed a continued growth since its beginning in 1955. From a traditional American one-man beginning the company has grown to its present size—having $200,000 authorized capital stock and having served clients as far south as Georgia and as far west as Wisconsin. On completion of the reorganization now in progress, it will have several new professional personnel and shall make available expanded services in market and business development as well as in technical project work.

GOULDS PUMPS, INC.

For those highly corrosive pumping jobs involving relatively small capacities—up to 75 g.p.m., Goulds Pumps, Inc., 229 Black Brook Rd., Seneca Falls, N. Y., now has available a 1-in. size Goulds-Pfaudler glassed pump. This unit will fill a demand in the chemical processing industries for a unit of lower capacities than available with the Model 3708 Goulds-Pfaudler glassed pump.

GREAT LAKES CARBON CORP.

*Low Density Mineral Filler*

A new mineral filler of extremely low density has been developed by Great Lakes Carbon Corp., a major supplier of filter aids and fillers. Called Dicalite Bulk-Aid, the new product is said to have the lowest apparent or bulk density of any known mineral filler. The product's light weight and bulking qualities, plus high brightness and excellent retention, make it especially suitable for use in paperboard. When used in dry powder formulations, Bulk-Aid effectively reduces bulk density and minimizes compaction caused by shipping and handling.

HERCULES POWDER CO.

*Overseas Plants*

Two Hercules Powder Co. overseas affiliates officially opened new plants within a three-day span this week. N. V. Hercules Powder Co., which maintains its offices in The Hague, dedicated its new chemicals plant at Zwijndrecht in The Netherlands on September 26. On September 29, Hercules Kemiska Aktiebolag, the Swedish affiliate, dedicated its new paper chemicals plant at Lilla Edet, Sweden.

The new plant at Zwijndrecht is designed to produce rosin size for the paper industry; rosin emulsifiers for the synthetic rubber industry; synthetic resins for protective coatings, adhesives, ink, floor coverings, and many other products for industries in Western Europe. Construction of this plant was begun in 1960, and it is expected that it will produce up to 30,000,000 lb. of resin derivatives a year.

Hercules Kemiska Aktiebolag's new plant is designed to produce a complete line of chemicals for the Swedish paper industry, although at the beginning of its operation, the Lilla Edet plant will devote its production entirely to the manufacture of regular and fortified rosin size. Construction of the Lilla Edet plant was begun in 1960, and the production of rosin size for the Swedish markets started in the summer of 1961.

Opening of the two new plants brings the total of production facilities owned by Hercules or their affiliates to 11 in seven foreign nations.

*Coating Division*

Two new managerial appointments in the Coatings Division of Hercules Powder Co.'s Polymers Dept. were announced. The new appointments are: **James R. Yeager,** assistant to the director of coatings materials sales and **Gregory N. Bruxelles,** manager nitrocellulose sales.

*Fiber Development Department*

The appointment of **William P. Crawley** as a sales representative in the Fiber Development Department of Hercules Powder Co. was announced here recently. Mr. Crawley will be working out of the department's Charlotte, N. C., office, located at 1214 Wachovia Bank Bldg., and his territory will consist of all of the southern states. The Hercules Fiber Development Department is concerned with the production and sales of polypropylene staple fiber and continuous multifilament yarns. The fibers are produced at the department's plant in Covington, Va., from resin supplied by Hercules.

HODAG CHEMICAL CORP.

**James Kuse** has been named director of market development by Hodag Chemical Corp., Skokie, Ill. Hodag manufactures surface active chemicals for a broad range of processing and manufacturing applications. Mr. Kuse will be responsible for the investigation of new markets for Hodag products, and he will aid the sales department in marketing new products.

S. W. HOOPER CORP.

An illustrated booklet, known as Bulletin C105, describing the new, improved K Series Cowan centrifugal pump screens, is now available from the manufacturers, S. W. Hooper Corp., Box 129, Dehman, N. Y.

The booklet describes the operation of both the "K" and the "KX" models and contains sectional elevations, exploded views, and drawings giving their general dimensions. Also included are charts listing information required when ordering, recommended motor speeds, and a suggested spare parts stock. In addition, there are installation notes, maintenance instructions, a description of construction details and performance curves.

HUYCK FELT CO.

*Benefit Dept.*

Huyck Felt Co.'s Benefit Department is entering its 50th year of employee service. Initiated in 1911 by Edmund Niles Huyck, the Benefit Department was established to do something for the many loyal employees who were obligated to stop work because of age, sickness, or incapacity. E. N. Huyck started his plan two years before the N. Y. State Workmen's Compensation Act became law. He also pioneered many industrial safety practices.

The original Huyck plan was designed to provide pensions and pay benefits in the event of disability or death. Despite the fact that it was available on a voluntary basis, over 98% of the employees subscribed. The cost of the benefit program was borne by both company and employees, with the company paying the major share. Operating deficits were made up by the company and surpluses were distributed to Benefit Department members.

Huyck Felt was one of the first small companies to adopt such a plan on a scientific actuarial basis. Through the years its effectiveness has increased and E. N. Huyck's spirit of social justice and his farsightedness have paid dividends for all. Not only have the workers been aided during periods of sickness and disability, but employee morale has been kept at a high level and the loss of skilled employees minimized.

I-T-E CIRCUIT BREAKER CO.

Completely new-design 4.16- and 13.8-kv. circuit breakers and switchgear—first in this rating developed specifically for stored-energy closing—are described in a new bulletin (No. 2800-2B) published by I-T-E Circuit Breaker Co., 1900 Hamilton St., Philadelphia 30, Pa. The three-color, 40-page bulletin provides drawings, sketches, photographs, specifications and charts as an aid to designers, architects and application engineers of industrial companies, utilities, contractors and consulting engineering firms.

JOHNS-MANVILLE

Users of metallic gaskets will find PK-152A a comprehensive source of authoritative information on "Johns-Manville Goetze Metallic Gaskets." Published by Johns-Manville, 22 E. 40th St., New York 16, N. Y., as a service to gasket users, this booklet is designed for maximum convenience and visual appeal. This 28-page book is divided into six basic sections—Spirotallic Gaskets, Metal Jacketed Gaskets, Flat Metal Gaskets, Corrugated Gaskets, Round Cross-Section Gaskets and Heavy Cross-Section Gaskets. Each section is fully illustrated with photographs and cross-sections, augmented by concise, descriptive copy.

MIDLAND-ROSS CORP.

*J. O. Ross Engineering Div.*

**George F. Schuning** was recently named general sales manager of the J. O. Ross Engineering Division, Midland-Ross Corp. One of the chief responsibilities in his new position is management of the eight Ross sales offices across the U.S.A.

J. O. Ross Engineering Division, Midland-Ross Corp. recently appointed **Gordon Stephens** as paper mill sales manager of the New York District Office. Mr. Stephens will coordinate and supervise the paper mill sales force located in the New York office. This office covers most of the mills in the Atlantic Coast Region of the United States.

MINERALS & CHEMICALS PHILIPP CORP.

The appointment of **William H. Somers** as Midwest Regional sales representative, and the reassignment of **W. E. Welliver** to Western Regional sales representative for the Paper Sales Department has been announced by G. A. Hale, manager of Paper Industry Sales for Minerals & Chemicals Philipp Corp., Menlo Park, N. J. Mr. Somers will assist O. W. Callighan in Michigan, and will also serve MCP's customers in Ohio, Indiana, Illinois, and Tennessee. Mr. Welliver will represent MCP in Wisconsin, Minnesota, and on the West Coast.

MINNEAPOLIS-HONEYWELL REGULATOR CO.

Three new specifications (S1004-1, S1004-2, and S1004-3) describe Honeywell's new PneumatiK Tel-O-Set line of instruments to supplement their existing miniature pneumatic instrumentation. The new complete line features automatic switching with no matching of any pressures; simple, accessible zero and damping adjustments; easy-to-read 4-in. charts with rectilinear recording, simple, calibrated control adjustments; optional front-set control adjustments; and easy-to-remove components. Complete details on the features, specifications and ordering information for individual models (ranging from single-pen or pointer noncontrol instruments to two-pen control stations for single station cascade systems) are available on these specifications.

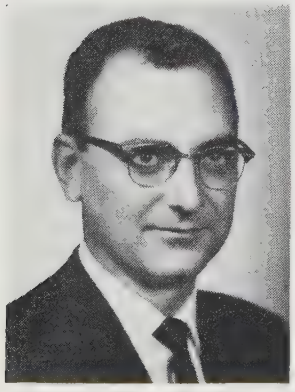
MORDEN MACHINES CO.

*Lava Tackle*

Development of a new lava tackle for the Morden Stock-Maker refiners is announced by Morden vice-president in charge of sales and services, John L. Sigler. The new lava fillings have been thoroughly mill tested and are recommended where maximum fiber development is required, as in the carbonizing, greaseproof and glassine field.

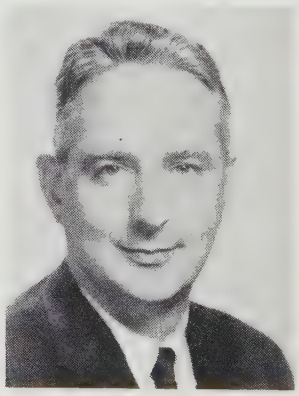
Production experience indicates that the Stock-Maker refiner with the new lava tackle gives fiber treatment comparable to a conventional stone roll multiple bedplate batch beater. These results are obtained at relatively moderate capital costs and low energy requirements per ton of production. In addition, the Stock-Maker refiner provides continuous production.

For detailed information write to Morden Machines Co., 3420 S. W. Macadam Ave., Portland 1, Ore.



*G. F. Schuning, Midland-Ross-Corp.*

*Gordon Stephens, Midland-Ross Corp.*



*John L. Sigler, Morden Machines Co.*

*Katharine A. Fralick, Morden Machines Co.*

*Staff*

Newly elected officers of Morden Machines Co., Portland, Ore., are **John L. Sigler,** who has been named vice-president, Sales and Services, and **Katharine A. Fralick,** who has been named secretary of the company.

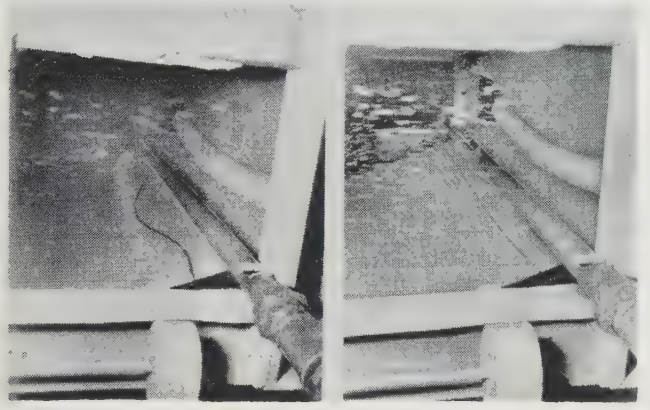
**Allen E. Carl** to the newly created post of application engineer it has also been announced.

*Allen E. Carl, Morden Machines Co.*

Mt. Hope Machinery Co.

Unretouched photos show installation of a Mount Hope Roll on a bottom wet felt at a major eastern paper mill. Left photo shows tendency to "cocking" of seam or guide line. It was taken immediately after installation of the roll before the machine was started up again.

The right hand photo shows the same position a few minutes later after the corrective action of the Mount Hope roll had straightened the seam. This is accomplished by rotating the



*Before* *After*

apex of the bow into the felt until the seam is straight. Then the roll is returned to its original position as shown. Since a felt running "cocked" will fill up faster and distort the weave, bringing the threads closer together and impairing drainage, the life of a felt—and its performance during its service lifetime—can be extended considerably by the use of the felt conditioning roll. Besides providing more uniform drainage across the full width of the felt, the Mount Hope roll also has the ability to prevent widthwise shrinkage throughout the lifetime of the felt.

Mount Hope rolls are engineered and fabricated by Mount Hope Machinery Co., Taunton, Mass.

Nichols Engineering & Research Corp.

The Board of Directors of Nichols Engineering & Research Corp., New York, N. Y., engineers and suppliers of equipment to industry and to the sanitary engineering field, have elected **H. Bach Nielsen,** general sales manager of the firm.

Noble & Wood Machine Co.

The Noble & Wood Machine Co., Hoosick Falls, N. Y., announces the appointment of Monarch Supply Co. (Division of Monarch Forge & Machine Works, Inc.), 2130 Northwest York St., Portland 10, Ore., as the exclusive West Coast sales and service representatives for Noble & Wood's complete line of stock preparation equipment (pulpers, jordans, jordan fillings, Tisco castings, refiners, beaters, agitators, etc.), wet board machines and laboratory handsheet machines.

The Monarch organization will cover the States of California, Idaho, Oregon, and Washington for Noble & Wood.

Nopco Chemical Co., Inc.

*Adhesive Additives FDA-Cleared for Use in Food Packaging*

Final regulations have been published by the Food and Drug Administration clearing paper defoamers and adhesive additives, produced by Nopco Chemical Co., for use in the manufacture of paper, paperboard and containers for the packaging, transporting and holding of food.

The list of products cleared on Aug. 30, 1961, includes 116 pulp and paper defoamers. Other miscellaneous products—wax and wax/rosin sizes, dispersing and wetting agents—are included in extensions granted by the FDA until Jan. 1, 1962.

On Sept. 12, 1961, the FDA cleared 42 Nopco defoamers, stabilizers, thickeners and resin emulsions; and 22 miscellaneous products—wetting and dispersing agents, stearate dispersions—as additives for adhesives used in food packaging. Five other adhesive-component products received extensions until July 1, 1962.

Further information and listings of the FDA-cleared products may be obtained by inquiry to Industrial Division, Nopco Chemical Co., Inc., 60 Park Place, Newark 1, N. J.

Oakite Products, Inc.

**Donald F. Moulton** has been appointed technical service representative in Vermont for Oakite Products, Inc., pioneer manufacturers of industrial cleaning and metal treating compounds.

Oilgear Co.

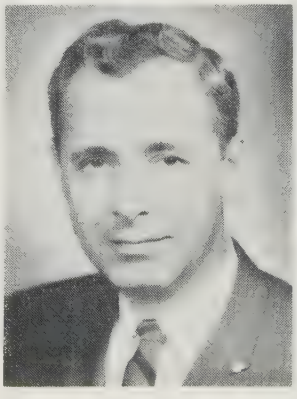
The Oilgear Co., 1572A W. Pierce St., Milwaukee 4, Wis., introduces its complete line of electrohydraulic servo controlled pumps and system components in Bulletin 47740. How the pumps and components work, what functions they perform, and where they are applied are described in detail.

Penick & Ford, Ltd.

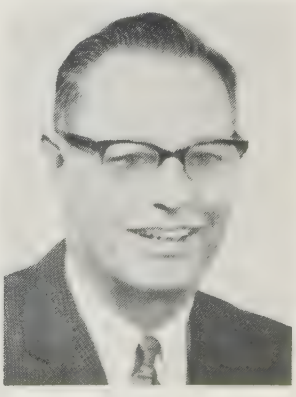
Available from Penick & Ford, Ltd. is a new booklet outlining a plan to help manufacturers convert their operations from bag handling to bulk handling of starch. Titled "Guide to Bulk Starch Automation," the booklet describes different systems, discusses their utility, and defines the engineering help P&F offers. It may be secured from the company at 750 Third Ave., New York 17, or any other P&F office.

*Appointments*

The promotion of **John K. Bonnell** to the position of paper mill representative of Penick & Ford, Ltd., Corn Refining Division, has been announced. Mr. Bonnell will call on paper and corrugating mills in Minnesota and Wisconsin.

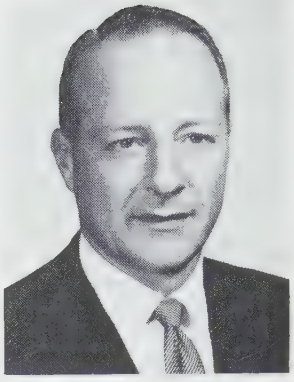


*John K. Bonnell, Penick & Ford, Ltd.*



*Frank Ware, Penick & Ford, Ltd.*

*A. B. Mindler, Pfaudler-Permutit, Inc.*



*Paul A. Fodor, Jr., Pittsburgh Plate Glass Co.*

The promotion of **Frank Ware** to the position of paper mill representative of Penick & Ford, Ltd., Corn Refining Division. Mr. Ware will call on paper and corrugating mills in Chicago, Wisconsin, and the Upper Peninsula of Michigan.

The appointment of **R. L. Post** as paper mill representative of Penick & Ford, Ltd., Corn Refining Division, has been announced. Mr. Post will service paper mills, bag plants, and corrugating plants in the Pacific Northwest.

Pfaudler Permutit Inc.

**A. B. Mindler** has been appointed to the newly-created post of manager of special applications sales for the Industrial Division of The Permutit Co., a division of Pfaudler Permutit Inc., manufacturers of ion exchange, water and waste treatment equipment and materials.

*Type M Precipitator*

The Permutit Division of Pfaudler Permutit Inc., 53 W. 43rd St., New York 36, N. Y., now has a six-page folder describing its new Type M precipitator, a unique sludge-blanket clarifier whose agitator arms are driven by water jets at the tips. The bulletin describes fully in illustrations and text the operation and design of the new solids contact precipitator. Operating graphs, based on field experience, show the Type M's quick accommodation to changes in raw water makeup, plus the results of a salt tracer detention test.

Pittsburgh Plate Glass Co.

Appointment of district sales managers for three West Coast district offices of Pittsburgh Plate Glass Co.'s chemical division has been announced. Appointed district managers for two newly-established sales offices were **H. Richard Sherburne** at Los Angeles, Calif., and **Arthur W. Wilson** at Portland, Ore.; also, **Donald A. Huff** at San Francisco, Calif., succeeding **O. W. Andrews** who was named director of marketing services with headquarters in Pittsburgh, Pa.

Appointment of **Paul A. Fodor, Jr.,** as director of sales for the chemical division of Pittsburgh Plate Glass Co., has also been announced by Chris F. Bingham, vice-president, sales—chemical division.

Pulp & Paper Know-How Associates

Since the widespread adoption of the policy of retirement at the age of 65, much has been said about the resulting waste of ability and experience. The Pulp & Paper Know-How Associates, Box 667, Holyoke, Mass., has been organized to offer to the industry the wealth of this experience and ability represented by a carefully selected group of outstanding men who have recently retired. Their combined experience covers most phases of the pulp and paper industry.

This organization does not propose to compete for the type of work now being well done by a number of consultants, but

***E. C. Tucker, a director; P. S. Bolton, president, Helen U. Kiely, secretary-treasurer; and C. H. Kent, vice-president and managing director of Pulp & Paper Know-How Associates***

rather does offer an experienced and very personalized outside viewpoint on the smaller but none-the-less important problems of every-day operation. It is believed that valuable assistance is available from members of this group in the fields of management, production, sales, product engineering, development, accounting, personnel procurement, industry mergers and many others.

Pyroxlyin Products, Inc.

Pyroxylin Products, Inc., of Chicago, has appointed **Jorge Willars** sales representatives of their manufacturing plant in Mexico D. F. The company produces lacquers, heat-sealing compositions, protective coatings, concentrates, and many special formulations.

Raybestos-Manhattan, Inc.

*Appointments*

**John McKinlay,** manager of Northeast District, has just been advanced to assistant manager Eastern Sales Region covering the sale of Industrial Rubber Products and Mechanical Packings for Raybestos-Manhattan, Inc., Passaic, N. J.

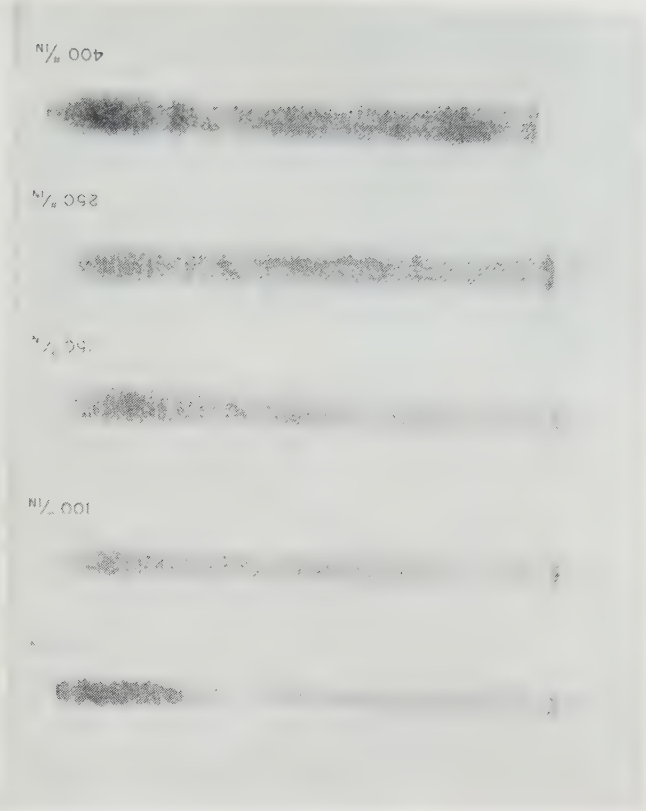
*Nip and Crown Roll Tester*

Manhattan Rubber Division of Raybestos-Manhattan, Inc., of Passaic, N. J., has a totally new approach to testing nips and crowns of rubber covered process rolls. Of special value in the maintenance of accurate nips and crowns of all rubber covered rolls, this test set is said to be the most effective and least costly method of assuring high quality runs, and of spotting trouble before it starts.

The test set, made up of 200 ft. of a specially developed carbon paper together with 200 ft. of white master sheet, is inserted in a shipping-dispensing carton with a serrated tearing edge. Included in the kit are complete instructions and a booklet discussing nips and crowns and other related information of value to the mill superintendent.

It is claimed that this test set overcomes the difficulties experienced with previously developed sets both in the storage of the test material and the finished test records. The completed record may be rolled, folded and stored any place to form a complete case history of service life.

This new unit is said to provide a completely accurate record at pressures of from less than 50 lb. to more than 1500 lb. per lineal inch. In addition to the obvious advantages of providing a carbon impression, the Manhattan nip and crown tester can be used for suction rolls to indicate any tendency to cup in the hole area, or to flake, crack, or corrugate.



***Impressions taken with the nip and crown roll tester***

A series of these tests taken over a period of time not only provides a good record of roll condition but also indicates in advance of failures the need for regrinding or other roll adjustment.

These kits are available through all Manhattan field offices or directly from company headquarters in Passaic, N. J. The price has been announced as $15 per unit and shipments are said to be made within 48 hr. of receipt of order.

The accompanying figure shows typical samples of impressions made with a wide variety of roll pressures.

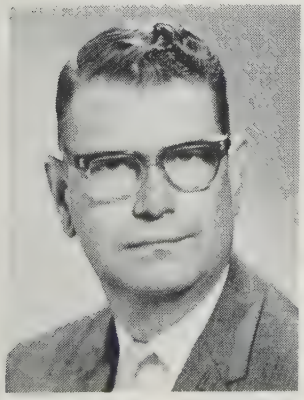
RESEARCH-COTTRELL, INC.

Research-Cottrell, Inc., Bound Brook, N. J., announces the appointment of **William C. Osborne** as vice-president in charge of engineering and research. Mr. Osborne, who also becomes a member of the Board of Directors of the corporation, will be responsible for all engineering and research activities of Research-Cottrell, one of the country's leading industrial air and gas cleaning manufacturers.

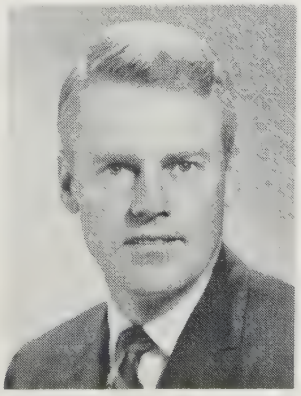
RICE BARTON CORP.

**Philip A. Peterson** has been appointed sales manager, special products, of Rice Barton Corp., Worcester, Mass., it was announced by Charles S. Barton, president. Mr. Peterson has been a key figure in sales since joining Rice Barton in 1951 and was recently named chief engineer in charge of trailing blade coater ranges. He will continue to be responsible for the sales of coating machinery as well as flash-drying plants and all new products.

**Carl J. Peterson,** who has been specializing in the sales of flash drying plants, has been appointed assistant sales manager.



***Philip A. Peterson, Rice Barton Corp.***



***Carl J. Peterson, Rice Barton Corp.***

ROOTS CONNERSVILLE BLOWER DIV.

Just published by Roots-Connersville Blower, new Bulletin RAS-261 presents the company's line of rotary positive displacement blowers (Type RAS) and gas pumps (Type RGS). The brochure outlines the advantages of these heavy-duty machines, including positive volume displacement, inherent operating flexibility that saves horsepower, high volumetric efficiency, low mechanical friction, adaptability for many gases, compactness, ability to deliver oil-free air or gas, and to handle moderate amounts of entrained liquids. Pictures, illustrations, and diagrams give full data on performance and operating characteristics. The Bulletin details the complete line of 36 sizes with capacities available from 1500 to 30,000 c.f.m. working pressures to 12 psig, and dry vacuums to 15 in. Hg. Copies of Bulletin RAS-261 can be obtained from Roots-Connersville Blower Div. of Dresser Industries, Inc., 900 W. Mount St., Connersville, Ind.

SANDOZ, INC.

**Raymond J. Mitchell** has been promoted to sales manager for the New York District Sales Office, Sandoz, Inc. and **Harvey G. Gendreau** succeeds Mr. Mitchell as sales manager of the Cincinnati District Sales Office.

DAVID J. SAUNDERS

**Daniel J. Saunders,** former vice-president of Pfaudler Permutit, Inc., has announced the opening of an office at 70 Pine St., New York 5, N. Y. Mr. Saunders will engage in a wide range of management, sales and other consulting work for companies manufacturing engineered products. In addition, he will act in a consulting capacity to organizations desiring technical services, where surveys, specifications, and recommendations are desired covering water and waste problems.

SPROUT, WALDRON & CO.

*Rotary Knife Cutter*

Sprout, Waldron & Co., Inc., announces the availability of an Advance Specification Sheet No. 215 on their improved 18-in. rotary knife cutter. This unit is designed to accept material in lump form through a wide throat hopper or in strip form through variable speed feed rolls. Other advantages in construction and design are discussed.

SQUARE D CO.

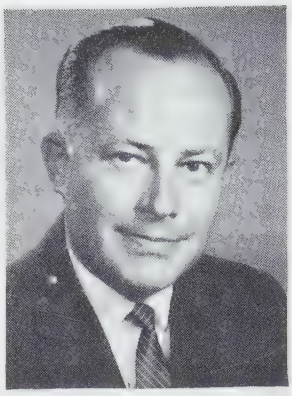
*Line Coupler*

A new line coupler which permits safe and easy removal of circuit breaker and starter equipment has been added to the Square D Spin Top enclosure for hazardous locations. It makes it unnecessary to disconnect the main power feeder to remove a unit. The branch connectors are fully insulated for complete safety to personnel. Hot lines cannot come into contact with any part of the enclosure. For details, write Square D Co., Dept. SA, 4041 N. Richards St., Milwaukee 12, Wis.

A. E. STALEY MFG. CO.

Expansion plans for the A. E. Staley Manufacturing Co.'s chemical sales organization at Decatur were announced by L. E. Doxsie, vice-president of the Decatur corn, soybean, and

chemical processing concern. "Though corn and soybean-derived products have long figured in the company's sales," Mr. Doxsie said, Staley's entry into the synthetic polymer field in 1959, the development of new chemical products, and our growth in this area since have called for an expansion and realignment of our chemical sales effort."



*Theodore P. Malinowski, A. E. Staley Mfg. Co.*

**Theodore P. Malinowski** has been named to head the reorganized chemical sales activity, effective Oct. 1, 1961.

TAYLOR INSTRUMENT COS.

*Magnetic Flowmeter*

The Taylor MAG-PIPE* electromagnetic flowmeter provides an output signal of 1–5 ma d.c. which may be transmitted with $^1/_2$% accuracy up to 10,000 ft. to space-conserving miniature receivers or controllers on central control panels. The flowmeter is ideally suited to many of the viscous liquids and suspensions found in the chemical, food, paper, plastics, rubber, and sewage treatment industries. It will accurately measure fluids with low velocities and with electrical conductivities as low as distilled water. For more information write for Bulletin 98418. Taylor Instrument Cos., Rochester 1, N. Y.

*Cylinder Operated Valves*

Light weight, fast-acting actuators have been combined with rugged ductile iron bodies in the new Taylor 1000V Series of cylinder-operated valves. Valves are provided for either two or three-way operation. The rated service pressures are 300 or 500 p.s.i., and the valves are available for cold or hot hydraulic service up to a maximum temperature of 400°F. Write for Bulletin 98419, Taylor Instrument Cos., Rochester 1, N. Y.

TESTING MACHINES, INC.

*TMI Reference Manual*

In the October issue of TAPPI the availability of the TMI Reference Manual for Quality Control and Research was mentioned. Although the reference manual cost $6.00 per copy to produce as indicated in the release it will be distributed free to qualified technicians and purchasing executives.

*High and Low Temperature Dynamic Tester for Plastics*

The TMI Maxwell Dynamic Tester for plastics shows the dynamic mechanical properties of plastics over wide frequency and temperature ranges for R&D and to help improve product engineering. The Maxwell machine was developed in the Princeton University Plastics Laboratory as a joint research project for the Navy and Army Signal Corps.

The magnitude of the strain can be controlled, making it possible to test over any desired portion of the stress-strain curve. It applies a sinusoidally oscillating strain to the test specimen.

The basic theory, certain test results and conclusions of Professor Maxwell are covered in detail in ASTM Bulletin 215. An unabridged reprint and an illustrated product specification sheet are available from Testing Machines, Inc., 72 Jericho Turnpike, Mincola, N. Y.

TEXAS GULF SULPHUR CO.

**H. Newton Cunningham** has been named assistant sales manager of Texas Gulf Sulphur Co., according to an announcement by A. Nelson Myers, vice-president and manager of sales.

U. S. STONEWARE CO.

The move of its engineering laboratories to a newly-acquired building at 2664 Gilchrist Road, Akron, Ohio, is announced by The United States Stoneware Co. Formerly occupied by a manufacturing division of a heavy industrial equipment firm, the modern 84,000 sq. ft. plant provides considerably increased floor space for the laboratory and permits much more efficient layout and working conditions.

WHITING CORP.

**A. R. Downing** has been named supervisor of development for Swenson Evaporator Co., a division of Whiting Corp., Harvey, Ill.

**A. C. Patsavas** succeeds Mr. Downing as manager of Swenson's Spray Dryer Department.

## *General*

ASTM

The name of the American Society for Testing Materials was officially changed to the American Society for Testing *AND* Materials with the signing of a court decree on Sept. 18, 1961, amending the Society's Charter originally granted in 1902 by the Commonwealth of Pennsylvania.

ASTM now has almost 11,000 members including individuals, companies, government agencies, and educational institutions. There are in addition about 7000 prominent engineers and scientists who serve as representatives of company members on the Society's 87 technical committees.

FIBRE BOX ASSOCIATION

**Lloyd Merwin,** vice-president of Crown Zellerbach Corp., and general manager, Gaylord Container Div., St. Louis, Mo., was elected president of the Fibre Box Association at the group's 22nd Annual Meeting in New York City. **Edwin D. Dodd,** vice-president, Owens-Illinois Glass Co. and general manager of its Forest Products Division, Toledo, Ohio, was elected vice-president and **William J. Cassady, Jr.,** vice-president and general manager of the Container Div., Mead Corp., Cincinnati, Ohio, was re-elected vice-president.

FOREST PRODUCTS RESEARCH SOCIETY

New dates for the 1962 Annual Meeting of the Forest Products Research Society which will be held at Spokane, Wash., have been announced. June 17–21, 1962, is the new date, replacing the earlier announced date of June 24–28. The 4-day meeting will feature technical sessions on all phases of the wood products industry, and plant tours. The event will draw an international attendance of more than 600.

SOCIETY OF PLASTICS ENGINEERS

To recognize outstanding accomplishments in plastics, the Society of Plastics Engineers has established an international award for fundamental contributions in plastics science and engineering. Comprising a gold medal, cash honorarium of $1000, and certificate, the award will be made for the first time at the 18th SPE Annual Technical Conference in Pittsburgh, Pa., Jan. 30-Feb. 2, 1962.

SOCIETY OF RHEOLOGY

*Bingham Medal Award*

The Bingham Medal of the Society of Rheology, highest award of this scientific origanization, was conferred on William R. Willets of the Titanium Pigment Corp.

Mr. Willets was honored at the 32nd annual meeting of the society held at the University of Wisconsin. The citation

* Trade Mark.

was read by John H. Dillon, president of the Textile Research Institute, and the presentation was made by John H. Elliott, of the Hercules Research Center in Wilmington, Del., president of the Society of Rheology.

Professor John D. Ferry, of the Department of Chemistry at the University of Wisconsin, was elected president of the society. He will take office at the conclusion of the meeting. Other officers were named also.

*William R. Willets, Titanium Pigment Corp.*

Rheology is the science of the deformation and flow of matter. It is applied in a wide variety of fields such as high polymers and plastics, metals, petroleum, rubber, paint, paper, printing ink, ceramics and glass, adhesives, floor preparations, and cosmetics. The society is a member society of the American Institute of Physics. Rheologists are drawn from education, industry, and government and represent many scientific disciplines.

Mr. Willets has been secretary-treasurer of the Society of Rheology since 1953 and was cited in the medal presentation for his strengthening of the funds of the society. He also was in charge of arrangements for rheology meetings in New York in 1949 and 1950.

He is a member of the Governing Board of the American Institute of Physics, a board member of the American Society for Testing Materials, past-chairman of the Pigments and Fillers Testing Committee of the Technical Association of the Pulp and Paper Industry, a fellow of the American Institute of Chemists, and a member of the American Chemical Society.

Born in 1905 in Montclair, N. J., he was graduated from the State College of Forestry in Syracuse, N. Y., with the B.S. degree and was employed successively by the Oxford Paper Co. and the Western Electric Co. He has been affiliated with the Titanium Pigment Corp., a subsidiary of the National Lead Co., since 1933 and is now assistant manager of the Technical Service Laboratories. During World War II he served as a consultant to the War Production Board on the conservation of paper resources.

His professional activities have covered much of the field of paper technology. During the past 30 years he has been the author or co-author of over 50 articles and technical papers. In recent years, he has been concerned with the rheology of papermaking and paper coatings. His work in this connection has contributed to theories of filler retention and has helped to acquaint the paper industry with rheological concepts.

### Water Pollution Control Federation

John G. Strange, president of The Institute of Paper Chemistry, Appleton, Wis., spoke before the 34th annual Water Pollution Control Federation meeting, Milwaukee, Wis., October 8–12.

As guest speaker at the Industry Day luncheon on October 11, Mr. Strange presented a talk, "A Rational Approach to Water Utilization." The talk was the culmination of the activities of part of the convention. The entire day was designated "Industry Day," concentrating the technical talks on the various aspects of industrial waste treatment. As a leader of the pulp and paper industry, one of the prime industries in this section of the country, Mr. Strange was the first choice to represent this important segment of American industry.

This year's Industry Day was co-sponsored with the Federation by the Institute of Paper Chemistry, the National Council for Stream Improvement, the Sulphite Pulp Manufacturers' Research League, and the Northwestern Pulp and Paper Association.

### Engineering Recruitment

"Is There a Future Scientist or Engineer in Your Home?" is the title of a booklet just released by Battelle Memorial Institute, Columbus, Ohio. The booklet is offered as a primer to help parents and leaders of young people in the discovery and encouragement of the nation's future scientists and engineers.

The booklet outlines characteristics which may indicate an embryo engineer or scientist. It also emphasizes that, if he or she is not discovered early enough and encouraged to complete required courses in high school, the changes are he will find it difficult to succeed at the university of his choice. The booklet lists the subjects and courses which are "musts" in high school. In addition, it suggests other academic and associated activities which help to develop the embryo engineer or scientist.

Commenting on the new booklet, B. D. Thomas, Battelle's president, said: "It represents the best thinking of a number of educators, guidance counselors, psychologists, engineers, and scientists, and it is directed primarily toward parents. With the heavy demands on our educational system, many parents have a vital interest in helping their children to select a career and prepare for it. In our complex age of specialization, providing parental guidance and encouragement is not an easy matter—even for the most conscientious parents. We hope this booklet may help parents to help their children."

The booklet further covers such subjects as (1) where to seek specific counsel, (2) possible financial aid for the college-bound youngster, and (3) possibility of a student working his way through college.

Single copies are available free (written requests only, please). Less than 100 copies, 10¢ each; up to 500, 9¢ each; up to 1000, 8¢ each; above 1000, 7¢ each. Requests should be in multiples of 10 and addressed to Public Services Office, Battelle Memorial Institute, Columbus 1, Ohio.

### Fiber Tester

A new high speed tester capable of determining the tensile properties of single fibers at elongation rates in excess of 1 million % per min. has been developed by Fabric Research Laboratories, Dedham, Mass., for Celanese Corp. of America, Summit, N. J.

Until now the choice of standard laboratory tensile test conditions has been based primarily upon the capabilities of available test instruments. That is, either slow tensile strains are applied by using equipment such as an Instron tensile tester run at speeds of the order of 1 in. per min., or equipment of the other extreme, namely ballistic impact testers with velocities ranging up to 2000 ft. per sec. are used. In the first instance test conditions approximate the use of a fiber after it is in a fabric or finished garment; the latter is suitable for simulating use conditions of body armor, impact on parachute shrouds, safety belts, and the like.

One area of fiber use which has received little attention is that of fiber processing; the conversion through successive stages from fiber to web to yarn and finally to fabric for articles of commerce. Processing velocities are in the range of 5 to 150 ft. per sec. For example, the surface velocity of a carding machine is 50 to 150 ft. per sec.; or in weaving, normal shuttle velocities are about 35 to 40 ft. per sec., while other textile processes such as winding and warping subject fibers to velocities of 5 to 50 ft. per sec.

It is obvious that the properties of fibers must be studied at these strain rates in order to understand and interpret the effects of processing velocities. The significance of the new tester is that it operates within the range of velocities and strain rates that fibers are subjected to during processing from the fiber through to the finished fabric stage.

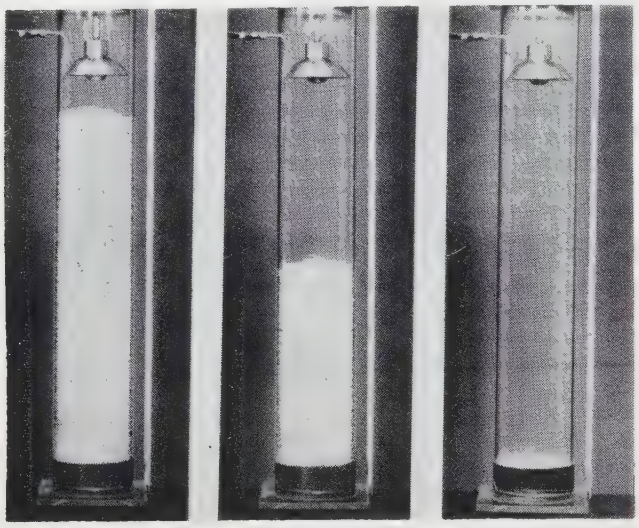
The basic design of the equipment consists of a gas-driven piston with a crystal force transducer for a load cell, and has incorporated in the system an oscilloscope for recording the information.

The tester has proved useful for examining viscoelastic properties of fibers as a function of testing rate, particularly at the combinations of higher strain level and short time experienced in processing which are not obtainable by conventional testers.

### Defoaming with Sound Waves

Industry is turning to a new sound wave system to destroy the massive quantities of foam now handicapping production of soaps, shampoos, beer, wine, soft drinks, milk, drugs, plastics, paper, petroleum, sewage disposal, chemicals, and hundreds of other products.

The Teknika system, for which patents are pending, shoots sound waves from a new type of sonic transducer "gun." The high intensity sonic energy actually explodes the undesirable foam within a special resonant chamber that is installed in production pipe-lines wherever the foam is formed.



***Sonic energy from the transducer "gun" at the top of the 3-ft. column destroys foam instantly. The second photo was taken 0.04 sec. and the third photo was taken 0.08 sec. after the first. This proves that a 3-ft. column of foam can be destroyed in less than a second***

Purely by the physical action of the sound, the system destroys foam at rates as fast as 1000 c.f.m., which is typical of some of the more serious foam-generating problems.

Gulf States Paper Co., Tuscaloosa, Ala., has installed a pilot Teknika continuous defoaming system specially designed for eliminating black liquor foam. Such foam is an exceptionally hard-to-handle problem in the southern kraft paper pulp industry. Teknika black liquor sonic defoaming systems prevent pollution of rivers and streams. In addition, the systems recover otherwise lost material, which is a source of valuable by-products. The new defoaming systems reduce offensive odors in pulp making.

Dewey & Almy Div. of W. R. Grace & Co., Cambridge, Mass., has installed on its paper coating machines a specially-developed pilot Teknika sonic defoaming system. This new system continuously defoams synthetic paper coating emulsions at the rate of 500 g. per hr. The resultant coated paper is completely free from uncoated areas, "fisheyes," and other defects previously caused by foam. No chemical additives are required.

### Improved Standard Observer for Colorimetry

The National Bureau of Standards recently completed a study of the variability in the spectral tristimulus values of the standard observer system for colorimetry. A statistical model was used in the study to obtain values for both "within-observer" and "between-observer" variability at wavelength intervals throughout the visible spectrum. These data may be used to determine the significance of differences between color measurements made by direct observation, and those achieved indirectly with the standard observer system.*

Since 1931, the International Commission of Illumination has recommended a standard observer for color measurement.† This observer consists of a table of values which are the averages of the measured quantities of the three primary colors (red, green, and blue) used by a group of human observers to arrive at a color match when viewing a mixed field. In 1959, an international committee on colorimetry proposed values for the standard observer, which in general are much higher in the short-wave end of the spectrum and somewhat higher in the long-wave end, based on data from observers who viewed an enlarged field in making their color matches.‡ However, neither of these sets of data includes the variability inherent in such measurable quantities. The present study was therefore undertaken by I. Nimeroff, J. R. Rosenblatt, and M. C. Danneniller of the Bureau staff, to develop an improved standard observer.

In the study, the average color-matching data of Stiles and Burch§ for 53 observers and the average-deviation data of Speranskaya‖ for 27 observers were analyzed to obtain between-observer variability. This variability was computed in terms of both variances and covariances; that is, the dispersion of the data around the mean of the observer values for the three primary colors was taken into account, as well as the interaction between the observer values for these colors. Stiles-Burch standard-deviation data for two observers who repeated their observations several times were analyzed to obtain within-observer variability.

The 80 sets of data analyzed in the study had originally been obtained by means of a visual tristimulus colorimeter. In this instrument, an observer views a split field, the halves of which are side by side, and by adding or subtracting the amounts of light emanating from the three different light sources (red, green, and blue) of the device, he arrives at a color match. Two of the primary colors are mixed to form one part of the field, and the third color is mixed with a fixed amount, $E_\lambda$, of a spectral test color, $T_\lambda$, of wavelength $\lambda$, to form the second field. The three adjustable primaries, whose wavelengths are 645.2, 526.3, and 444.4 m$\mu$, respectively, may be denoted $R$, $G$, and $B$, and their amounts $R_\lambda$, $G_\lambda$, and $B_\lambda$, so that the condition of color matching may be expressed as:

$$E_\lambda T_\lambda = R_\lambda R + G_\lambda G + B_\lambda B$$

Empirical amounts, $R_i$, $G_i$, and $B_i$, for the $i$th observer in a series of observers, may then be represented by means of this mathematical model developed in the present study:

$$\begin{aligned} R_i &= R + b_{Ri} + \epsilon_{Ri} \\ G_i &= G + b_{Gi} + \epsilon_{Gi} \\ B_i &= B + b_{Bi} + \epsilon_{Bi} \end{aligned}$$

where $R$, $G$, and $B$ are the averages for the values of all the observers; $b_{Ri}$, $b_{Gi}$, and $b_{Bi}$ are an observer's biases (that is, characteristics of one observer that make his observations different from all the others); and $\epsilon_{Ri}$, $\epsilon_{Gi}$, and $\epsilon_{Bi}$ are the observer's errors.

The standard observer proposed in 1959 for colorimetry utilizes a linear equation to transform the system of real primaries ($x$, $y$, and $z$). In the present study the transformation was treated as arbitrary, that is, as one in which only the variances and covariances in the real-primary system are *different from* zero, while the variances and covariances that involve the constants of the transformation *are* zero.

Hence, with the model formulated above, the variances and covariances could be determined in terms of the real primaries, and the values thus found could be substituted in the arbitrary transformation to determine the variances and covariances in the imaginary-primary system. Values for the between-observer and within-observer variabilities ar-

rived at in this manner were then tabulated at 10 mμ-wavelength intervals.**

The improved standard observer, which includes means, variances, and covariances, will aid investigators in determining the extent to which an observer with normal color vision tends to make matches that are different on his successive attempts, or are different from those made by other normal observers.

* For further technical details, see Variability of Spectral Tri-stimulus Values, *J. Research NBS* 65A (Phys. and Chem.) No. 6 (1961).
† Proceedings of the eighth session, Commission Internationale de l'Eclairage, Cambridge, England (Sept., 1931), pp. 19–29.
‡ Detailed report of progress (Sept., 1957, to April, 1959) of the CIE Working Committee W-1.3.1, Colorimetry, by Deane B. Judd.
§ N.P.L. colour-matching investigation: Final report (1958), by W. S. Stiles and J. M. Burch, *Optica Acta* 6: 1 (1959).
‖ Determination of Spectral Color Coordinates for 27 Normal Observers, by N. L. Speranskaya, CIE Committee Report, and, in part, NPL Symposium 8, Her Majesty's Stationery Office, London, 1, 317 (1958); also Optics and Spectroscopy VII, 424 (1959), (OSA Translation).
** These tables are included in reference 1.

# OBITUARIES

## Elbert Haven Neese

Elbert H. Neese, Sr., Chairman of the Board of Beloit Iron Works and an officer of the company for 45 years, died suddenly at Beloit, Wis., on September 17.

Mr. Neese was born in Monroe County, W. Va., on Aug. 14, 1886. He graduated from Goldey College, Wilmington, Del.

In 1903 he joined the Pusey & Jones Corp. and was appointed a vice-president and sales manager in 1912. He became vice-president of Beloit Iron Works in 1916, its president in 1931, and board chairman in 1952.

At the time of his death he was director and board chairman of Sandusky Foundry & Machine Co.; past director of Warner Electric Brake and Clutch Co.; Beloit Foundation, Inc.; and the Beloit State Bank.

He left two sons, E. H. Neese, Jr., president of Beloit Eastern Corp., Downingtown, Pa.; Alonzo H., vice-president and treasurer of the Beloit Iron Works; and a daughter, Mrs. Elizabeth Teague. His wife Laura, who is a well-known water color painter, survives.

Mr. Neese became a member of the Technical Association of the Pulp and Paper Industry in 1916.

## Theodore M. Gilbert

Theodore M. Gilbert, president of the Gilbert Paper Co., died suddenly Sept. 17, 1961, at his summer cottage, Waupaca, Wis.

Mr. Gilbert had been affiliated with the Gilbert Paper Co. for 38 years, joining the company in June, 1923, following graduation from the University of Wisconsin. He had been active in all phases of the business and had served as secretary, treasurer, and vice-president before his election as president in 1954. He had been a director of the company since 1927.

Mr. Gilbert was born in Neenah, Wis., on July 6, 1900. He was active in industry and community affairs; serving as a trustee of the Theda Clark Memorial Hospital, chairman of the Menasha Planning Commission, director of the Pulp Consumer Association, and a member of the Executive Committee of the Writing Paper Association.

He is survived by his wife, Ruth; three sons, Charles N. of Menasha, Theodore M., Jr., and Albert D., both of Neenah; and seven grandchildren.

## George W. Mead

George W. Mead, 90, honorary chairman of the Board and former president of Consolidated Water Power & Paper Co., died at his home in Wisconsin Rapids, Wis., on Oct. 2, 1961. Mr. Mead served as company president from 1916 to 1950.

Mr. Mead was well known, not only as an industrialist but also as a civic leader and philanthropist.

He was born Feb. 22, 1871, in Chicago, the year of the great fire in that metropolis. He attended schools in Oak Park and Rockford, Ill., graduating from Rockford High School in 1888. After one year at Beloit College, he transferred to the University of Wisconsin, receiving his degree—bachelor of arts—with the class of 1894. He married Ruth Emily Witter of Wisconsin Rapids on Oct. 18, 1899.

For eight years following his graduation from the University, Mr. Mead was engaged in the furniture business at Rockford with his brother, D. Ray Mead. But the untimely death of his father-in-law, J. D. Witter, called him to Wisconsin Rapids in 1902 to assist in managing Witter's business affairs. Mr. Witter and Nels Johnson had been prime movers in organizing Consolidated Water Power & Paper Co. in that year.

Typical of Mr. Mead's leadership was the start-up of the Wisconsin Rapids mill in 1904 with the first electrically-driven paper machines in the industry. In 1933 under Mead's leadership the company acquired the Massey coating patents, laying the groundwork for taking all Consolidated mills out of newsprint production. After extensive experimentation, the industry's first successful high-speed coater built into a paper machine went into operation in Wisconsin Rapids in 1935.

After suffering a stroke in September, 1950, from which he never fully recovered, Mr. Mead resigned from his many business affiliations and has been living in retirement at his winter home in Miami Beach, Fla., and his summer home in Wisconsin Rapids. He is survived by two sons, Stanton, Wisconsin Rapids, and Walter, Oconomowoc; a daughter, Mrs. Emily Baldwin, Wisconsin Rapids; and nine grandchildren.

## Martin J. O'Leary

Martin J. O'Leary, technologist in the Paper Section of the National Bureau of Standards, Washington, D. C., passed away on October 2, at the age of 68. Mr. O'Leary had served the Government for 33 years, having joined the Bureau staff after 20 years of experience in the paper industry. The author of 14 scientific publications, he was well known for his research on techniques for manufacturing new types of papers, especially those made entirely from glass fibers. Mr. O'Leary had received many awards and commendations for superior performance, including the Department of Commerce medal for meritorious service. He had been a member of TAPPI up to the time of his retirement.

## Theodore W. Dunn

Theodore W. Dunn, founder of the Dunn Paper Co., was born Oct. 9, 1872, in Osborne, Ohio, and passed away on October 18 in Port Huron Hospital after an illness of several weeks. He celebrated his 89th birthday just 9 days before his death.

Theodore Dunn, at the age of 16, started his paper career as a laborer at the Detroit Sulphite Pulp & Paper Co. He succeeded his father, George Friend Dunn, as manager and was vice-president of the Detroit mill when he resigned in 1922 to start his own company.

The Dunn Paper Co., located in Port Huron, Mich., started

operating in 1924. Theodore Dunn was active in its management until a year ago. He was president of this company from its beginning until his recent retirement to chairman of the board and treasurer. George C. Dunn succeeded his father as president of the company.

Mr. Dunn's wife, Isabel Curtis Dunn, passed away in 1952 while attending a paper convention in New York City. She and Theodore Dunn were known to many in the paper industry. He held life memberships in a number of the industry associations. Though he lost interest in most material things during the last few months of his life, he maintained his keen interest in the mill and the paper industry until the end.

Mr. Dunn is survived by a son, George C. Dunn, president and secretary of the firm, and a daughter, Mrs. George Burton Smith, both of Port Huron; a grandson, Dr. Charles A. Chidsey III, Bethesda, Md., a granddaughter, Mrs. James B. Felton, Springdale, Utah, and three great-grandchildren.

Mr. Dunn joined the Technical Association of the Pulp and Paper Industry in 1915 as a charter member.

## Frank X. Hayes

Frank X. Hayes, vice-president and director of the Noble & Wood Machine Co., Hoosick Falls, N. Y., died Tuesday, Oct. 24, 1961, at the Mary McClellan Hospital, Cambridge, N. Y., after a short illness.

Mr. Hayes was born in Hoosick Falls, N. Y., on Oct. 2, 1892. He was employed by the Noble & Wood Machine Co. for the past 40 years and will be remembered by many of the old time papermakers.

He was a veteran of World War I and served in a medical detachment. He is survived by his wife, two sons, five daughters, six grandchildren, and two great grand-children.

Mr. Hayes became a member of the Technical Association of the Pulp and Paper Industry in 1948.

## Henry W. Schmidt

Henry W. Schmidt, vice-president in charge of manufacturing of the Upson Co., Lockport, N. Y., died on Oct. 23, 1961, after an illness of two months.

Mr. Schmidt graduated from the Wharton School of Finance, University of Pennsylvania, in 1928 and from Harvard Law School in 1932. He practiced law in Elizabeth, N. J., from 1932 to 1940 and was a trial examiner before the National Labor Relations Board, Washington, D. C., from 1937 to 1940.

He joined the Upson Co. as assistant to the president in 1941 and became vice-president in 1949.

He is survived by his widow, the former Jeanne A. Upson, a daughter of one of the founders of the company; a daughter, Suzanne, and son, Henry W., Jr.

Mr. Schmidt became a member of the Technical Association of the Pulp and Paper Industry in 1952.

## Henry B. Greenwood

Henry B. Greenwood, president and general manager of Greenwood Engineering Co., Inc., Baltimore, Md., died in July.

Mr. Greenwood was born in Kent County, Md., on Sept. 8, 1898, and joined the F. X. Hooper Co., Inc., Baltimore, Md., in 1920 as vice-president in charge of manufacturing. In 1945 he became president and general manager of Greenwood Engineering Co., manufacturers of corrugated container making machinery.

He became a member of the Technical Association of the Pulp and Paper Industry in 1953.

## James Cheney Kimberly

James C. Kimberly, retired, former vice-president of Kimberly-Clark Corp., died in October.

Mr. Kimberly attended the Massachusetts Institute of Technology, after which he became general superintendent of pulp and paper mills for Kimberly-Clark Corp. He retired in 1954.

Mr. Kimberly became a member of the Technical Association of the Pulp and Paper Industry in 1917.

## August C. Orthmann

August C. Orthmann, president and director of the Orthmann Laboratories, Inc., Milwaukee, Wis., consulting and analytical chemists, died recently.

Mr. Orthmann joined the U. S. Glue Co., Milwaukee, Wis., as a chemist in 1902. In 1908 he became chemist for Pfister & Vogel Leather Co. In 1931 he took over the Pfister & Vogel Laboratory to form the Orthmann Laboratories, Inc. He published many technical articles in the *Journal of the American Leather Chemists Association* and in trade papers serving the leather industry. He laboratory tested much of the wood pulp passing through the Port of Milwaukee.

He became a member of the Technical Association of the Pulp and Paper Industry in 1931.

## William Gordon Fowler

William G. Fowler, technical representative of Riordon Cellulose Ltd., London England, died in July in London.

Mr. Fowler was born in Newry, Ireland, on Jan. 19, 1903, and received his early education in Montreal, Canada.

He served his traineeship at Laurentide Co., Ltd., Grand'-Mere, Que., St. Maurice Valley Paper Co., Montreal, and Price Bros. Co., Quebec. From 1932 to 1940 he was a technical representative for Bowaters Mersey Paper Mills in England and joined British International Paper Ltd. in 1940. This company became Riordon Cellulose Ltd.

Mr. Fowler became a member of the Technical Association of the Pulp and Paper Industry in 1947.

## Abraham Lewinstein

Abraham Lewinstein, vice-president and technical director of Appleton Coated Paper Co., Appleton, Wis., died in Appleton on Oct. 20, 1961.

Mr. Lewinstein was born in Kovarsk, Lithuania, on April 3, 1901, and came to the United States with his mother in 1912. He graduated as a chemical engineer from the University of Minnesota in 1926.

He joined the Appleton Coated Paper Co. in 1926 as a chemist. He became chief chemist in 1931, technical director in 1948, and was elected vice-president in 1957.

During his years of research with Appleton Coated Paper Co. he gained world-wide recognition for his work on off-machine coating. Much of his research concerned the development of new uses for colored and white coated papers. He was responsible for the introduction of a number of pigments used in colored papers. He was also closely connected with the development of many coated specialties for which his firm is known.

He is survived by his widow; one son, Simon, a student at the University of Wisconsin; a brother, Samuel of Duluth, Minn.; and two sisters in Duluth.

He became a member of the Technical Association of the Pulp and Paper Industry in 1930 and has taken an interest in the work of the TAPPI Coating Committee over the years.

# DIVISIONS AND COMMITTEES

## *Authorized Committee Assignments*

The following new committee assignments were made during October, 1961. The total number of assignments now active in the 64 TAPPI technical committees is about 400. An identifying number is given by TAPPI Headquarters to each work assignment made by a committee chairman and approved by the appropriate division chairman. Assignment numbers should be requested for all activities of technical divisions.

| *Assignment no.* | *Committee* | *Title and objective* | *Committeeman in charge* |
|---|---|---|---|
| | | COATING AND GRAPHIC ARTS DIVISION | |
| 1016 | Divisional Activities | "Liquid Flow Phenomena." | J. G. Curado and C. G. Albert |
| | | ENGINEERING DIVISION | |
| 4060 | Operations Research | "An Economic Model of the Paper Industry—A Preliminary Study." In the preliminary study, the subcommittee is to seek expert opinion on the feasibility of obtaining a comprehensive economic model of the United States or North American Pulp and Paper Industry. The ultimate objective would be the development of such a model. | R. R. Hodges |
| | | TESTING DIVISION | |
| 8032 | Chemical Methods | "Alkali Solubility of Pulp T 235 m-58 and ICCA 2:61." International adoption of the method for alkali solubility of pulp. | |
| 8033 | Wax Testing | "Tensile Strength of Petroleum Wax." Test procedure to determine the resistance (in pounds per square inch) of a wax specimen to fracture when being elongated. | R. L. Murphey |
| 8034 | Wax Testing | "Flow Tests." Develop standard tests. | M. V. Hunter |
| 8035 | Wax Testing | "Composition of Waxes." Tests to determine composition. | Fred Brill |
| 8036 | Wax Testing | "Nomenclature and Definitions." Organization of Symposium on characterization of petroleum waxes. | G. A. Weisgerber |
| 8037 | Wax Testing | "Surface Wax Determination." To prepare a method for the determination of surface wax of waxed paper. | Gene Unmuth |
| 8038 | Wax Testing | "Friction." To develop a suitable test method for the measurement of friction of waxes and/or waxed paper. | R. C. Paris |
| 8039 | Wax Testing | "Odor of Petroleum Wax." To obtain tentative standard status for the above test. | J. D. Tench |
| 8040 | Wax Testing | "Gloss of Waxed Paper." To establish a satisfactory method for measuring the gloss of waxed paper which will correlate with visual glass. | R. C. Paris |
| 8041 | Wax Testing | "Blocking Paint of Petroleum Wax." To review and edit the present method and advance the method to a tentative standard. | J. Walker |
| 8042 | Wax Testing | "Modulus of Rupture of Petroleum Wax." Test procedure to determine how much weight a wax bar can support under specified loading conditions before the wax bar fractures. | J. E. Turner |
| 8043 | Wax Testing | "Congealing Point of Petroleum Wax, Including Petrolatum." Test method to determine the temperature at which a molten wax specimen no longer will flow. | G. A. Weisgerber |
| 8044 | Wax Testing | "Drop Melting Point of Petroleum Wax, Including Petrolatum." Test method to determine the temperature at which a solid wax specimen becomes fluid. | G. A. Weisgerber |
| 8045 | Wax Testing | "Melting Point (Plateau) of Petroleum Wax." Test method to determine the melting point of certain petroleum waxes. | G. A. Weisgerber |
| 8046 | Wax Testing | "Needle Penetration of Petroleum Wax." Test method to determine the hardness or consistency of petroleum waxes. | K. G. Arabian |
| 8047 | Wax Testing | "Cone Penetration of Petrolatum." Test method to measure the firmness, consistency, or mobility of petrolatum. | K. G. Arabian |
| 8048 | Wax Testing | "T 636 Oil Content of Petroleum Waxes" (ASTM D 721–56 T). (1) Editorial revision; (2) improve precision. | D. R. Cushman |
| 8049 | Wax Testing | "Peroxide Number of Petroleum Wax" (ASTM D 1832–61 T). Publication by TAPPI as tentative method. | L. C. Crews |
| 8050 | Wax Testing | T 650 sm-55 "Oxidation Resistance of Paraffin Wax." Withdrawal from publication by TAPPI. | |

## *Corrugated Containers Division*

### *Raw Materials Committee*

Meeting was called at 2:20 p. m., Sept. 5, 1961, in San Francisco, Calif.

Mr. Wilmer advised of his inability to find time to continue as chairman, and of the Steering Committee's appointment of A. L. Magnuson as new chairman. He indicated his willingness to work as part of a subcommittee group as his time allows.

A. Magnuson reported that the Steering Committee plans to establish a uniform terminology for the different activities of the industry by asking subcommittee chairmen to assemble a list of terms concerned with each activity, to be defined at a later date. The collection of terms and their definition will be on a continuing basis. He asked that the lists of terms be submitted to him by Jan. 1, 1962. H. Adickes will advise all subcommittee chairmen of this responsibility.

A. Magnuson also told of the concern on the Steering Committee of lack of order in the auditoriums while papers were being read. Subcommittee chairmen are asked to support the quieting of such interference.

He related that the Steering Committee has set up a chart showing how the programming of future meetings will be constructed. This will delegate responsibility for selection of papers to be presented at future meetings and should stimulate projects and publication of papers, and should make for smoother programming. Committee chairmen will be advised.

R. M. Morris, Jr., stated that there is a need for standards acceptable for mill and converter goals.

W. Hurrell said that his company set up standards of operating conditions, and when uniform manufacturing conditions on the corrugator are established, then changes of adhesives make-up may be tried. He further said that when the moisture content of liner is below 5%, his plant experiences difficulty and must compensate with steam showers.

C. Robson contributed that in his plant operation, glue clearances are checked on every shift.

Chairman Robson commented on the importance of the committee embarking on a serious project, relating that the industry is in need of progressive development, and is pressed for the need of cost reduction in production. He indicated that part of the responsibility of the committee is to work to establish specifications meaningful to mill and corrugator.

G. H. McGregor asked about the value of a collection of observations on ranges of moisture content which gives a range of adaptability.

W. Hurrell asked about the possibility of some determinations of effective moisture content and water absorption as a helpful guide to corrugators. He also asked for a method of measuring stretch and tensile of medium. He also indicated a desire for a standard method of specifying characteristics for liner and medium.

R. M. Morris, Jr., pointed out that the Concora test does not indicate ease of runability, or runability, but rather that it does represent strength potential.

G. H. McGregor wishes the committee to make some reports and to that end will conduct a survey among committee members.

H. Adickes and F. Albert will canvas the committee for suggestions for new items for the agenda. They will report on this at the February meeting.

The fall 1962 program, at Boston, will be two days. The program for the National Convention, February 1962, will be one day, probably Wednesday, February 21.

The proposed Coatings and Additives Committee, under H. Johnston, is still under consideration.

C. Robson gave a report on his Liner and Medium Subcommittee meeting, held earlier in the day. He told about the problems of this committee in selecting a project for action. All present agreed on the importance of this committee and its relationship with improved production.

HERBERT J. ADICKES

## *Medium and Liner Subcommittee*

Chairman Robson opened the meeting at 9:15 a.m., Sept. 5, 1961 in San Francisco, Calif.

C. Robson distributed report of a study of loose edges on heavy board. He related the problems in adhesion of components of liner and medium.

R. M. Morris, Jr., said he is still trying to find a good single test for runability, pointing out that his mill production aims at producing a balanced sheet. In medium he feels that major characteristics are: basis weight, Concora, and moisture. Secondary characteristics are: Cobb, tensile, etc.

C. Robson said that from a containermaker's viewpoint, there is a need for specifying, to mill suppliers, the quality needed.

F. Albert feels that because of the use of reclaimed stock, mills are unable to control finished characteristics of medium, which he believes to be the source of difficulty in getting uniform production performance.

# *Testing Division*

## *Chemical Methods Committee*

The Chemical Methods Committee met Aug. 16, 1961, at the Hotel Queen Elizabeth, Montreal, Que., Canada.

*Intrinsic Viscosity.* In the minutes of the last two meetings a method for determining intrinsic viscosity has been described in which the specimen is dissolved in CED in an Ubbelohde viscometer by agitation with purified nitrogen. It was pointed out, however, that several dilutions in the same viscometer would mean that the different concentrations would be run at different velocity gradients, which would give rise to error in the extrapolation. All viscosities should be determined at the same velocity gradient. It was also mentioned that the various uncertainties, such as average D.P., D.P. distribution, velocity gradient, and others, might lead one to question the relative importance of velocity gradient.

Work on intrinsic viscosity is continuing.

*Pentosans.* A method based on the Swedish orcinol procedure has been approved, with a few changes, by the Standards Committee.

*Molecular Chain Length Distribution.* This method has been approved by the Chemical Methods Committee and is being placed in a different format at the suggestion of officers of the Testing Division.

*Carboxyl.* Companion methods for the determination of carboxyl, the sodium bicarbonate-sodium chloride method, and the methylene blue method, have been approved by the Chemical Methods Committee and by the chairman of the Standards Committee—with a few changes.

*Paraffin in Paper.* A method was prepared and approved by the Chemical Methods Committee with one negative vote. The chairman of the task group is investigating further before sending the method up through channels.

*Metals in Paper.* Methods for the determination of metals in cellulose and paper should be ready by February. The metals are Hg, Pb, As, Ni, Ca, Mg, Cu, Fe, and Mn.

*Melamine Resin.* A method for the determination of melamine resin in the presence of glue has been approved by the Chemical Methods Committee and forwarded through channels.

*Reducible Sulfur.* T 406, Reducible Sulfur in Paper, came in for considerable criticism, as it did at the February meeting. This method needs a complete revision, which would

***Chemical Methods Committee***

probably require about one man-year of laboratory work. Volunteers for this work are solicited.

*History of Alpha-Cellulose Methods.* A history of the methods for the determination of alpha-cellulose has been promised by next February.

## *Paper Shipping Sack Testing*

Chairman M. L. Taylor asked the committee for comments or corrections to minutes of the last TAPPI Paper Shipping Sack Testing Committee meeting Feb. 20, 1961. On motion duly seconded, it was *Voted:* to approve, without change, the Feb. 20, 1961, minutes of TAPPI Paper Shipping Sack Testing Committee meeting.

M. A. Burnston, technical assistant, TAPPI, New York, N. Y., addressed the meeting to offer his assistance on any problems that might arise concerning current or future TAPPI projects undertaken by the TAPPI Paper Shipping Sack Testing Committee.

Chairman Taylor thanked Mr. Burnston for his remarks, assuring him that the committee appreciated and would ask for his help if it was required.

### *Project No. 608. Procedure for Wet Tensile Testing of Wet-strength Shipping Sack Papers (1956)*

W. L. Shoemaker, chairman of the subcommittee responsible for this project, whose other members are G. P. Heller and R. P. Kessler, reported that the subcommittee was in agreement with the proposed revision of TAPPI method T 404 m-50 published in *Tappi,* June 1960, entitled "Tensile Breaking Strength of Paper and Paperboard."

He stated that his committee felt it should direct its attention toward developing a recommended procedure for treating the wet-strength shipping sack test specimens prior to making strength tests on the wet sheet.

Chairman Taylor stated that the problem of wet tensile testing of shipping sack papers was twofold: (1) how to prepare wet specimens reproducibly, and (2) running the test.

It was agreed that since the proposed TAPPI Standard T-404 should be used for dry as well as wet tensile testing, Project No. 608 should be considered cancelled: that the same subcommittee responsible for Project No. 608 should proceed with a recommended referee method of preparing test specimens of shipping sack paper prior to performing wet-strength test. This method should include: (1) wetting procedure, (2) blotting procedure, and (3) time interval between blotting and testing.

### *New TAPPI Project—"Method of Preparing Wetted Samples of Shipping Sack Paper for Wet-Strength Tests"*

Chairman Taylor advised the meeting that application would be sent to TAPPI for new project number covering "Method of Preparing Wetted Samples of Shipping Sack Papers Prior to Performing Wet-strength Tests."

Committee responsible for this project would be: W. L. Shoemaker, chairman, G. P. Heller, R. P. Kessler.

### *Project No. 769. Tensile Energy Absorption of Shipping Sack Paper (1958)*

Following subcommittee is responsible for developing a suggested method for Tensile Energy Absorption of Paper: H. S. Welsh, chairman, K. A. Arnold, M. L. Taylor, and D. M. Yost.

H. S. Welsh reported that in accordance with the minutes of the Feb. 20, 1961 meeting, copies of proposed TAPPI Suggested Method, Tensile Energy Absorption (Feb. 20, 1961), had been submitted to TAPPI Paper Testing Committee for balloting.

He stated that although there were sufficient ballots returned to approve the proposed suggested method, there were also enough comments to warrant a revision of the document which will be resubmitted to the TAPPI Paper Shipping Sack Testing Committee in the near future.

*Paper Shipping Sack Testing Committee*

Mr. Welsh furnished those present with copies of a revised draft of the proposed suggested method, dated Aug. 7, 1961. This draft, he said, will be further modified by comments just received from W. A. Wink, chief, Paper Evaluation Section, Institute of Paper Chemistry, Appleton, Wis.

Mr. Welsh advised that when the final revised draft is submitted to TAPPI for reballoting, copies would be furnished to the TAPPI Paper Shipping Sack Testing Committee.

There was discussion concerning the possible inclusion in the final draft of a preconditioning procedure for specimens to be tested.

Concensus was that details of sampling and of conditioning of samples should be included by reference to the appropriate TAPPI Standards. It the event that prior history of samples or details of conditioning *within the scope* of the applicable standard methods affect the test results, the existence of these extraneous effects should be recognized by means of appropriate explanatory notes describing their effects on test results.

Mr. Welsh reported that according to the Feb. 20, 1961, minutes of meeting, his committee was asked to develop a faster, less precise, routine control method of measuring tensile energy absorption for consideration by the Routine Control Methods Committee.

He stated that this method would be written in the near future and submitted to TAPPI; that he would, at that time, send a copy to each member of the TAPPI Paper Shipping Sack Testing Committee.

### *Project No. 770. Measurement of Coefficient of Friction of Shipping Sack Paper (1959)*

Subcommittee responsible for this project is: H. C. Martin, chairman, A. R. Ewing, and R. P. Kessler.

R. P. Anderson advised that in accordance with the minutes of TAPPI Paper Shipping Sack Testing Committee meeting, Feb. 20, 1961, he had asked H. C. Martin to replace him as chairman of this subcommittee since business commitments made it impossible for him to continue work on this project; that Mr. Martin had graciously accepted the responsibility of carrying the work of this project to completion.

Mr. Martin announced that since coefficient of friction of shipping sack paper is a fundamental property that should not be difficult to measure, he would like to proceed with the gathering of test data on existing instruments now being used.

Accordingly, Mr. Martin's subcommittee met briefly following the TAPPI Paper Shipping Sack Testing Committee Meeting and "decided to conduct an informal round robin

merely to determine the 'order of magnitude' of the differences in coefficients of friction as measured by the different commercial instruments and by several homemade instruments. It is hoped that this work can be completed prior to the February TAPPI meeting."

***New Business***

Chairman Taylor asked the meeting to comment on the advisability of initiating a new TAPPI project to measure moisture vapor transmission rates on various moisture barrier sheets used in paper shipping sacks.

Dr. Arnold advised that, since the P.S.S.M.A. Technical Committee was asked by the Packaging Institute Bag Committee to provide water-vapor permeability ratings on barrier sheets used in multiwall constructions, it might be advisable to wait for the completion of that work before introducing this subject to the TAPPI Paper Shipping Sack Testing Committee.

C. G. Peterman
*Asst. Executive Secretary*

## *Pulp Testing Committee*

The meeting was called to order by H. A Smith, chairman pro-tem, on Tuesday afternoon at 2:00 p.m., Aug. 15, 1961, in Montreal, Que.

The minutes of the February 21 meeting were read and approved.

Report on Projects

*No. 470. Pulp Consistency—Lyman C. Aldrich*

Herb Smith is going to contact Mr. Aldrich, who was not present, to determine if he is going to continue with this project.

*No. 636. High Freeness Pulps*

Chairman Smith suggested that since this project is inactive it should be dropped. Mr. Elsevier reported he was out to The Institute of Paper Chemistry recently. Here he observed they were nearing the end of work on an instrument that would handle pulps in all freeness ranges. Decided not to drop project.

*No. 751. Reference Pulp—C. E. Brandon*

Mr. Smith asked if anybody was interested in establishing another grade of reference pulp. The second round-robin on the western sulfite has been completed and Mr. Brandon is preparing a report for publication. This project has been completed.

*No. 848. Revision of T 213 m-43, Dirt in Pulp*—J. E. Ray, Physical Methods Committee, and J. d'A. Clark, Standards Committee

Mr. Clark is refraining from making a revision of this project until the revision of Dirt in Paper is completed. Mr. Clark's method was published in TAPPI about a year ago. It has not been submitted yet for a ballot vote.

*No. 850. Revision of T 200 m-45, Lab. Beater*—V. C. Setterholm, Physical Methods Committee, and Curt Walseth, Pulp Testing Committee

This project has been completed and reissued as T 200 m-60 as a Tentative Standard because of its low precision.

*No. 8014. Revision of T 233 sm-53, Fiber Length by Classification*—J. d'A. Clark, Standards Committee

The revised method has been submitted for a ballot vote to be completed by August 1. Mr. Clark could not report on the results of the vote because he has been away from home.

***Pulp Testing Committee***

*No. 8015. Blotter Specifications in T 205 m-58, Forming Handsheets for Physical Tests on Pulp*—J. d'A. Clark, Standards Committee

Dr. Clark's report under this assignment is to be prepared for publication in TAPPI.

Mr. Rastetter reported that groundwood pulp made from chips was difficult to couch off the sheet molt during the handsheet making process. Mr. Smith related from his experience in which he found it difficult to remove the sheet when the blotters became wet. Mr. Clark described a new couching method which facilitated sheet removal. Most committee members present believed the present couching method satisfactory but the blotters supplied by various manufacturers are not uniform in their absorption characteristics. There followed a discussion on what recourse should be followed in the event of receipt of unsatisfactory blotters. It was agreed that this probably would be an individual matter. Mr. Smith suggested that comments be forwarded to Dr. Clark.

*No. 8016. T 218 m-59, Forming Handsheets for Optical Tests of Pulp*—B. B. Thomas, Optical Properties Committee

The object of this project is to provide an improved alternative to T 218 for eventual replacements.

Mr. Wiles commented that Glens Falls Research of I. P. Co. used to form their optical sheets on the British sheet mold. Recently they have changed to the use of the Buchner funnel. However, he believes a more uniform sheet was formed on the sheet mold.

Mr. Wiles, as a member of the Optical Properties Committee, agreed to bring to that committee's attention the various differences observed between the two methods.

*No. 8022. Revision of T 224 sm-45, Ball-Mill Joint Task Force*—CPPA-TAPPI

Mr. Cameron of the CPPA heads this task force. Dr. Ramsay of the Brown Co. is a member. Leon Gervais of Canadian International Paper recently has asked to join this committee. Various methods are being studied. The task force hopes to have a method ready to report upon by the time of the September 1962 meeting of the Testing Conference.

Other Methods Under Jurisdiction of the Pulp Testing Committee

T 210 m-58, Weighing, Sampling and Testing Pulp for Moisture

This method was revised in 1958.

T 220 m-60, Physical Testing of Pulp Handsheets

The committee voted 14 to 0 in favor of the recent revision. Ten members did not vote.

T 221 m-51, Drainage Time and Drainage Factor of Pulp

This method requires committee action.

Mr. Elsevier reported that The Institute of Paper Chemistry is working on an instrument and a method that will cover the high freeness as well as the regular range of pulps.

Mr. Wiles reminded the committee that they still will have to vote on acceptance of such a method and therefore any action should not be made until the work is completed.

T 225 sm-60 Lab. Processing of Pulp—Kollergang Method

J. d'A. Clark has made corrections to this method and it has just been issued in loose-leaf form.

T 226 sm-52, Specific External Surface of Pulp

This method requires committee action. It will be held in abeyance however until the work done by the International Paper Co. is better known.

T 227 m-58, Freeness of Pulp

Work on this project has been completed.

T 231 sm-60, Zero-span Breaking Length of Pulp

J. d'A. Clark made corrections again in 1960 and the method has been issued in loose-leaf form.

It was brought to the committee's attention that a set of jaws designed by The Institute of Paper Chemistry is superior and gives improved results especially at forces above 12,000 p.s.i.

Dr. Clark will make a comparison of the designs to see if the jaws covered in the present method can be modified to give the same results as those of the Institute. Action to be taken will be considered at the February meeting.

T 232 sm-58, Fiber Length of Pulp by Projection

Dr. Clark plans to revise this method so as to utilize the technique presented in his paper on T 234 (item following). He will have the revised method ready by February 1962. Committee Assignment No. 8025 was given to this project.

T 234 sm-60, Coarseness of Pulp Fibers by Protection

Dr. Clark will revise this method and present it at the next meeting of the committee. Committee assignment No. 8026 was given to this project.

New Business

H. Smith indicated he would appreciate receiving any suggestions for a chairman of the Pulp Testing Committee.

C. Walseth was asked to perform but is unable to accept because he is the incoming chairman of the Pacific Section of TAPPI.

Mr. Wiles made the following report: There has been a general industry-wide dissatisfaction with the Valley beater test method (T 200), particularly as regards inability to make different beaters agree with each other, and in holding them in calibration. This perhaps has been recognized by the Pulp Testing Committee in their recent degrading of method T 200 to a tentative method. The Technical Practices Committee of APPA, with D. F. Morris as chairman, has appointed a study committee to look into this matter and to make recommendations for improvement. This study committee, with Mr. Wiles as chairman, has C. E. Brandon, B. K. Mayer, and H. A. Smith as additional members. They are working closely with Mr. Nethercut and Mr. Winchester of TAPPI.

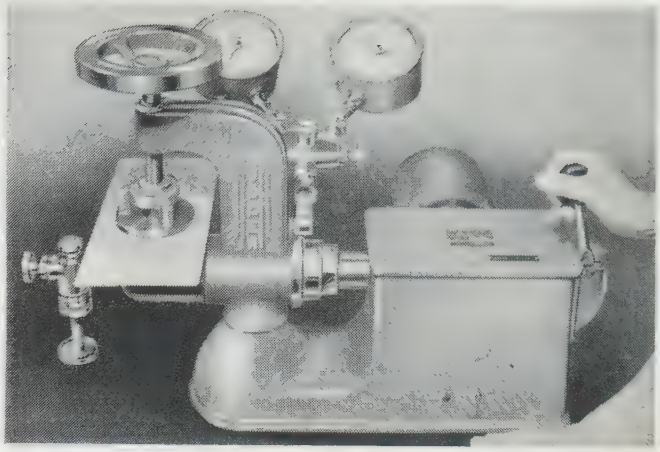
This study committee feels that the problem may be answered in one or the other of the following four fashions.

1. Accept the present Valley beater and its attendant TAPPI method as the best available and "live with it."

2. Modify the existent equipment and attempt to remedy its deficiency, keeping, however, the basic beater test principles.

3. Adopt as a substitute, or perhaps as another supplementary method, one of the presently available pieces of equipment—the Mead refiner, the PFI beater and the Jokro mill have been mentioned—and build a new method around one of them.

4. Develop a brand-new instrument and method.

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As of now, the committee feels that proposal (1) above is untenable and will look into the other three—2, 3, and 4—in that order.

The next meeting of the Pulp Testing Committee will be held on Tuesday morning of Feb. 20, 1962, at the Commodore Hotel in New York City.

G. E. Rabeier
*Secretary Pro-Tem*

## *Routine Control Methods Committee*

The meeting was called to order by Chairman L. J. Slentz in the Salon Chaudiere at 11:30 a.m., Aug. 26, 1961, at the Queen Elizabeth Hotel, Montreal, Que.

Chairman Slentz opened the meeting by giving a progress report of the committee's activities as follows:

| | |
|---|---|
| Methods under current consideration | 55 |
| Methods published | 9 |
| Methods ready for publication | 6 |
| Methods dropped | 2 |

He then stressed the importance of speeding up the process of reviewing, editing, and forwarding methods to TAPPI for publication. A backlog of unpublished methods at TAPPI headquarters insures a steady publication of the methods. To build up such a backlog, he recommended that methods 61-8 to 61-25 (Federal Test Methods) be sent to TAPPI, with a minimum amount of editing, for publication as currently written. He then stated that he would send to all members a complete listing of all unassigned methods. Each member to review the list and notify M. P. Boland which method he will accept for review and writing.

The 55 proposed methods under consideration were then reviewed and assignments made.

At 12:15 p.m. the meeting was adjourned.

B. Q. Haynes, *Secretary*

# LOCAL SECTION ACTIVITIES

## *Meeting Calendar*

***January, 1962***

5 Central District, Empire State Section, University Club, 431 Fayette St., Syracuse, N. Y. Panel meeting. "On Machine Clay Coated Board."

8 Bay District, New England Section, Armand Restaurant, Framingham, Mass.

9 Lake States Section, Green Bay Elks Club, Green Bay, Wis. "Waste Wood Utilization." Visit to mill of Green Bay Paper & Pulp Co., Green Bay, Wis.

9 Metropolitan District, Empire State Section, Stouffer's, 100 E. 42nd St., New York, N. Y. "Newsprint from Deinked Fiber," by John Rich, Garden State Paper Co.

9 Chicago Section, Chicago Bar Association, 29 So. La Salle St., Chicago, Ill. "Evaluating and Controlling Properties of Printing Papers."

9 Ohio Section, Manchester Hotel, Middletown, Ohio. "Coating Processes and Technology," by George Booth, Dilts Machine Co., Fulton, N. Y.

11 Northern District, Empire State Section, Hotel Woodruff, Watertown, N. Y. Students' night.

12–13 Gulf Coast Section, Tuscaloosa, Ala.

16 Berkshire District, New England Section, Country Club of Pittsfield (Mass.). "Trends in Beating and Refining," by Charles Vickery, E. D. Jones Corp., Pittsfield, Mass.

16 Pacific Section, Bellingham, Wash. "Engineering."

16 Indiana District, Ohio Section, Marott Hotel, Indianapolis, Ind. "Patents and the Technical Man," by H. R. Woodward, Patent Attorney.

16 Golden Gate Section, San Francisco, Calif. Talk by W. F. Linke, American Cyanamid Co.

18 Eastern District, Empire State Section, Queensbury Hotel, Glens Falls, N. Y. "Executives Night."

18 Kalamazoo Valley Section, Hotel Harris, Kalamazoo, Mich. Paper Makers Get-Together.

19 Southeastern Section, DeSoto Hotel, Savannah, Ga.

***February, 1962***

1 Lake Erie, Cleveland Engineering Society, 3100 Chester Ave., Cleveland, Ohio. "Industrial Psychology."

2 Central District, Empire State Section, University Club, 431 E. Fayette St., Syracuse, N. Y. "Papermill Engineering," by K. G. Taylor, Eastern Engineering Co., Atlanta, Ga.

6 Chicago Section, Chicago Bar Association, 29 So. La Salle St., Chicago, Ill. "Testing Instruments Used for Paperboard, Cartons and Corrugated," by George Maltenfort, Container Corp. of America, Chicago, Ill.

7 Western District, Empire State Section, Crown and Anchor Restaurant, Niagara Falls, N. Y. "Plastics in the Paper Industry."

8 Ohio Section, Manchester Hotel, Middletown, Ohio. "Computer Control for the Paper Industry," by Charles Carroll and David Kirk, I.B.M. Co.

8 Northern District, Empire State Section, Hotel Woodruff, Watertown, N. Y. "Headbox Hydraulics."

13 Indiana District, Ohio Section, Marott Hotel, Indianapolis, Ind. Education Meeting. City High School Teachers.

15 Eastern District, Empire State Section, Queensbury Hotel, Glens Falls, N. Y. "Consistency Control"—Panel Meeting.

***March, 1962***

1 Kalamazoo Valley Section, Hotel Harris, Kalamazoo, Mich. "Developments in the Graphic Arts."

1 Lake Erie, Plant tour.

2 Central District, Empire State Section, University Club, 431 Fayette St., Syracuse, N. Y. "Instrumentation of Paper Machines," by L. L. Thompson, I.B.M. Research Center, Yorktown Heights, N. Y.

6 Chicago Section, Chicago Bar Association, 29 So. LaSalle St., Chicago, Ill. "Foamed Plastics as a Packaging Medium."

7 Western District, Empire State Section, Crown and Anchor Restaurant, Niagara Falls, N. Y. "Stream Improvement."

8 Northern District, Empire State Section, Hotel Woodruff, Watertown, N. Y. Junior Award Papers.

12 Bay District, New England Section, Armands Restaurant, Framingham, Mass.

13 Ohio Section, Student Center, Miami University, Oxford, Ohio. "New Fibers from Wood," by Herman Mark, Polymer Research Institute, Brooklyn, N. Y. Joint meeting with Indiana District and students and faculty of Miami University Paper School.

13 Metropolitan District, Empire State Section, Stouffer's, 100 E. 42nd St., New York, N. Y. "Problems for Research," by K. A. Arnold, St. Regis Paper Co., New York, N. Y.

13 Lake States Section, Mead Hotel, Wisconsin Rapids, Wis. Panel discussion on "Paper Finishing Operations." Visit to mill of Nekoosa Edwards Paper Co., Port Edwards, Wis.

15 Eastern District, Empire State Section, Queensbury Hotel, Glens Falls, N. Y. "High School Essay Students Night."

16–17 Southeastern Section, Wilmington, N. C. Visit to mill of Riegel Paper Corp., Acme, N. C.

20 Pacific Section, Camas, Wash.

20 Indiana District, Ohio Section, Marott Hotel, Indianapolis, Ind.

20 Berkshire District, New England Section, Wendell Sherwood Hotel, Pittsfield, Mass. "Study of Fibrous Structure," by A. H. Nisson, Rensselaer Polytechnic Inst., Troy, N. Y., and "Effect of Fibrous Structure on Paper Properties," by W. Galley, E. B. Eddy Co., Hull, Que., Canada.

***April, 1962***

2 Pioneer Valley District, New England Section, Storrowtown Tavern, W. Springfield, Mass. "Printing."

3 Chicago Section, Chicago Bar Association, 29 So. La Salle St., Chicago, Ill. "Electronic Applications—Corrugated."

4 Western District, Empire State Section, Crown and Anchor Restaurant, Niagara Falls, N. Y. Visit to plant of Hazel Atlas Div., Continental Can Co.

5 Kalamazoo Valley Section, Hotel Harris, Kalamazoo, Mich. "Chemi-Mechanical Pulping."

5 Lake Erie Section, Cleveland Engineering Society, 3100 Chester Ave., Cleveland, Ohio.

6 Central District, Empire State Section, University Club, 431 E. Fayette St., Syracuse, N. Y. Senior night and Junior awards.

10 Metropolitan District, Empire State Section, Stouffer's, 100 E. 42nd St., New York, N. Y. "Reclaimed Fiber."

10 Lake States Section, Conway Hotel, Appleton, Wis., C. J. West Memorial Meeting. Tour of facilities of The Institute of Paper Chemistry, "What Should Research be Doing for the Pulp and Paper Industry?"

12 Ohio Section, Manchester Hotel, Middletown, Ohio. "Scientific Approaches to Paper Making," by Jasper Mardon, Oxford Paper Co., W. Carrollton, Ohio.

12 Northern District, Empire State Section, Hotel Woodruff, Watertown, N. Y. "Wet-Strength Resins for Paper."

12 Joint meeting with Beaver Falls Chapter A.I.C.

17 Indiana District, Ohio Section, Marott Hotel, Indianapolis, Ind. "Research"—Panel Meeting.

19 Eastern District, Empire State Section, Queensbury Hotel, Glens Falls, N. Y.

27 Delaware Valley Section, Riegel Ridge Community House, Milford, N. J. Visit to mill of Riegel Paper Corp., Milford, N. J. 2 p.m. Dinner Meeting. 6:30 p.m. "New Concepts in Testing Wet Pulp," by O. Hovland, Riegel Paper Corp.

***May, 1962***

1 Chicago Section, Chicago Bar Association, 29 So. La Salle St., Chicago, Ill. "Electro-types, Plate Making—New Developments."

1 Golden Gate Section, San Francisco, Calif. R. M. True Memorial Lecture by K. Sarkanen, Finland.

4 Central District, Empire State Section, University Club, 431 E. Fayette St., Syracuse, N. Y. Dinner-dance.

5 Western District, Empire State Section. Dinner-dance.

10 Ohio Section, Hartwell Recraction Hall, Cincinnati, Ohio. Dinner-dance.

10 Northern District, Empire State Section, Woodruff Hotel, Watertown, N. Y. Dinner-dance.

11–12 Gulf Coast Section, Edgewater Gulf Hotel, Edgewater Park, Miss. Annual meeting.

15 Berkshire District, New England Section, Country Club of Pittsfield, Mass. "The Rogers' Story," by George Archer, Rogers Corp., and "The Dexter Tale," by Allan Johnson.

15 Indiana District, Ohio Section, Marott Hotel, Indianapolis, Ind. "The Printing Process—Part II. Offset Lithography."

15 Lake States Section, Holiday Inn, Madison, Wis. "Forest Genetics." Visit to Forest Products Laboratory.

17 Pacific Section, Gearhart Hotel, Gearhart, Ore. Annual meeting. Joint meeting with Pacific PIMA.

18–19 Virginia-Carolina Section, Canton, N. C. Visit to mill of Champion Papers, Inc.

19 Eastern District, Empire State Section. Annual Meeting.

25 Metropolitan District, Empire State Section. Ladies' Night. Tavern on the Green, Central Park, New York, N. Y.

25–26 Southeastern Section, Wanderer Motel, Jekyl Island, Ga.

26 Lake Erie Section, Cleveland, Ohio. Annual meeting.

***June, 1962***

1–2 New England Section, Chatham Bar Arms, Chatham Bar, Mass.

7–9 Empire State Section, Whiteface Inn, Whiteface, N. Y. Maine-New Hampshire Section. (To be announced).

19 Indiana District, Ohio Section, Anderson Ind. Country Club. Fifth Annual Golf Outing.

## *New England (Bay District)*

The Bay District of the New England Section of TAPPI held a technical meeting at Armands Beacon Terrace, Framingham, Mass., on Sept. 11, 1961, with approximately one hundred members and guests in attendance.

The technical program consisted of a paper by Dr. J. W. Vanderhoff, Physical Research Laboratory, Dow Chemical Co., Midland, Mich., entitled "The Mechanism of Film Formation of Latexes."

In his talk Dr. Vanderhoff made use of films and slides which showed the actual formation of latex films under varying conditions of several types of latex.

Following the presentation of Dr. Vanderhoff's paper, a very lively question and answer period was held.

L. Paul Crane, *Secretary*

## *Ohio*

A meeting of the Ohio Section of TAPPI was held on September 12 at the Manchester Hotel in Middletown, Ohio.

Paul J. McLaughlin of the Rohm & Haas Co. of Philadelphia was the meeting speaker, his subject being "Contribution of Acrylic Polymers to Paper Products." An abstract of his talk follows.

The acrylic polymers are based principally on the esters of acrylic acid or of methacrylic acid and are supplied to the paper industry usually in the form of aqueous emulsions of excellent mechanical stability. These are used primarily in pigmented coatings (as binding agents to hold pigments to the paper surface) or in saturation of paper (to improve initial and internal tear strength, toughness, and extensibility).

The process of coalescence by which the discrete particles of the dispersion are brought together to form films of polymer is basic to any application.

In the case of pigment binding, because of the extremely high pigment volume content used in the industry, the polymer regions are probably discontinuous "spotwelds." In order to get adequate adhesion comonomers which bond well to cellulose. A polymer specially designed for this use is Rhoplex $\beta$-15. Where printing demands are less severe, Rhoplex B-60A is also useful.

Acrylic polymers for saturation of cellulose or synthetic fiber papers have been "tailored" to provide a variety of property combinations. For example, high internal (trapezoid) tear strength and good "bond" fabriclike feeling are given by Rhoplex B-25. Where good solvent resistance with a balance of tear and tensile strength are needed, Rhoplex HA-18, Rhoplex HA-12, and Rhoplex B-15 are useful. "B-stage" papers undergoing cure to a thermoset condition are attainable when Rhoplex AC-200 or Rhoplex AG-201 are used as saturants and the resulting papers give excellent adhesion to wood or metal and superior outdoor durability. As a class, the acrylic polymers show outstanding resistance to degradation by heat and ultraviolet radiation.

R. E. Essex, *Recording Secretary*

## *Delaware Valley*

The Delaware Valley Section of TAPPI held its first meeting of the year on Sept. 21, 1961, at Lamb's Tavern in Spring-

field, Pa. The program for the day consisted of a tour through the newly-built Research and Engineering Center of Scott Paper Co. at Philadelphia, Pa. The tour was followed by a meeting at Lamb's Tavern. The honored guests were Loren V. Forman, vice president of research and engineering, James J. Eberl, assistant vice-president of research, A. S. Erspamer, manager of specialty papers, and Quentin C. Weaver, assistant director for administration; all employees of Scott Paper Co.

The speakers for the evening were P. H. Hersey, director of technical service at P. H. Glatfelter Co. and Martin Silverburg of International Business Machine Corp. Mr. Hersey spoke on "Developing a Wet-End Control Program" while Mr. Silverburg spoke on the future outlook of process control with topic "Application of the Computer to Wet-End Control."

PETER N. YIANNOS, *Secretary*

## *Kalamazoo Valley*

On October 5, the Kalamazoo Valley Section of TAPPI held its first meeting joint with the Deinking Conference. Approximately 220 people were in attendance and the evening speaker was J. D. Dailey, Sr. vice-president of Allied Paper Co.

HARRY B. PARKER, *Secretary*

## *Lake States*

***September***

The Lake States Section of TAPPI opened its 1961–62 season Friday, September 29, with a Fun Day gathering at the North Shore Golf Club in Menasha, Wis. Approximately 120 members and guests turned out to play golf, trap shoot, and attend the dinner in spite of a cold rainy day. Prizes were awarded in golf and trap shooting after the dinner in the evening.

***October***

The second meeting of Lake States Section of TAPPI was held Tuesday, October 17, at the Wausau Club in Wausau, Wis. Plant tours of the D. J. Murray Manufacturing Co. and Wausau Iron Works were conducted in the afternoon. There were two tours at each plant starting at 1:30 and 3:00 p.m. Approximately 75 members toured both plants.

R. W. Theisen, sales manager of the Products Division of the Wausau Iron Works, and J. R. Haller, chief engineer of the D. J. Murray Manufacturing Co., both gave very interesting talks on the history of their respective companies to 115 members of TAPPI and guests.

F. G. PAVLICK, *Secretary*

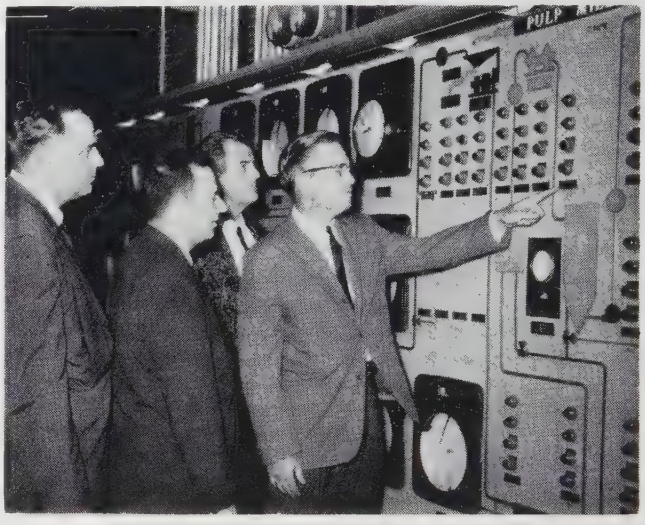
## *St. Louis*

The St. Louis Section of TAPPI resumed their meetings September 21 with a special mill tour of Alton Box Board Co. in Alton, Ill.

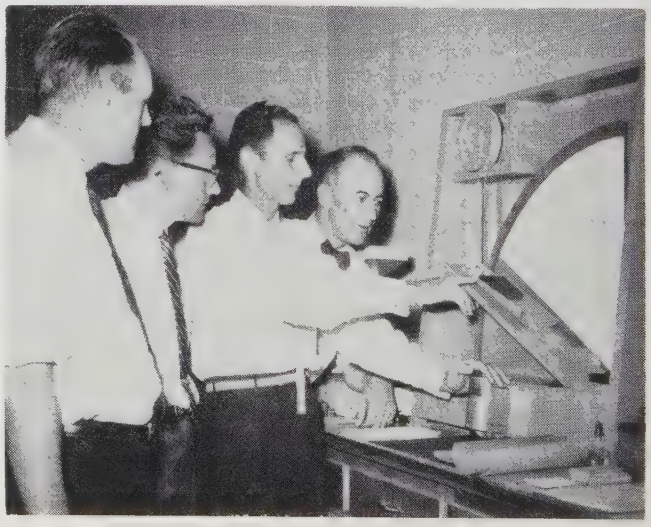
Over 90 members and guests attended the dinner meeting and tour. Featured speaker of the evening was Robert E. Bowen, vice-president in charge of mill production of Alton Box Board Co. Mr. Bowen's presentation gave a background of the Alton mill and its current operation related to paper and boardmaking. After the dinner meeting and speaker, the group was given a guided tour of the Alton Box Board mill production and laboratory control facilities. Attending members and guests had the opportunity to observe the entire process of paper- and boardmaking from pulp to finished product and board laminations.

Special guest of the evening was True D. Morse, assistant to the president of Alton Box Board Co., former undersecretary of Agriculture.

*R. E. Bowen, Alton Box Board Co.; William J. Rice, chairman, St. Louis Section; True D. Morce, Alton Box Board Co.; and B. L. Scallet*




*At pulp mill control board—Alton Box Board Co., R. W. Long and D. A. Pohlig, Packaging Corp. of America; Gordon Fisher, Minnesota & Ontario Paper Co.; and George Searles Alton Box Board Co.*



*At the Product Control Laboratory—Alton Box Board Co. Lew Heslep, Morningstar-Paisley, Inc.; Gerald Tyrer, Dow Corning Corp.; N. Schumaker, Alton Box Board Co.; Richard Koehrmann, International Shoe Co.*

*At the rewinder-Alton Box Board Co. Barry Scallet, Anheuser-Busch Co.; C. L. Eletson, Hopper Paper Co.; True D. Morse and Chet Hausman, Alton Box Board Co.; and J. R. Scohy, Westinghouse Electric*

*Officers and Executive Committee of the Western District, Empire State Section. Front row: R. Vaeth, Program vice-chairman; R. Zimsmeister, membership chairman; L. B. Timmerman, membership vice-chairman; and S. Fleming, program-member. Rear row: H. B. Hulse, chairman; W. I. Arnold, treasurer; and C. A. Kremers, vice-chairman. Missing from the above photograph: N. I. Johnson, secretary, F. Heil, program-chairman, W. Russel, arrangement-chairman, and O. Anderson, Jr., attendance-chairman.*

## *Empire State (Central District)*

On Oct. 6, 1961, a dinner meeting was held by the Central District, Empire State section of TAPPI, at the University Club of Syracuse, in Syracuse, N. Y.

The speaker for the October 6 meeting was N. R. Baldwin of Kamyr, Inc., Hudson Falls, N. Y. Mr. Baldwin explained Kamyr's position as basically being machinery designers and more or less consultants to the paper industry rather than being process chemical engineers and machinery builders. By handling the pre-engineering package of equipment, piping, and instrumentation, Kamyr supplies their customers with a package ready for operation, thereby maintaining the quality of the investment as well as the quality of the product produced. Mr. Baldwin presented a general discussion on the various types of Kamyr continuous cooking units available. Although his company is perhaps best known for its work in the high quality bleachable grade kraft field, there have been a number of important steps to produce pulps of lower strength requirements and by other pulping processes. Mr. Baldwin noted that although there are 65 units in operation or on order around the world today, among these units are some operating on neutral sulfite semichemical both for corrugating grades and for bleached grades, one on cold soda in South America, and several operating on materials other than wood chips such as the agricultural residues. Kamyr has also made a break-through in the sulfite field with the installation of a sodium-base bisulfite unit in central Europe. Flow sheets of each of the various types of units were displayed and explained by Mr. Baldwin, showing the major components of these systems. As a starting point in understanding the operation of the equipment, Mr. Baldwin went into some detail on the explanation of principles of operation and the equipment used for the high quality unit commonly in use in North America.

One of the more interesting developments discussed was the ability to do brown stock washing following time at temperature in the digester vessel before pressure release. We understand that Kamyr has recently accepted an order in Canada for a unit designed for 325 tons per day designed with this principle included, and that a unit in Australia is already performing very satisfactorily on a soda based cook using eucalyptus wood.

Following Mr. Baldwin's discussion there was a general question-and-answer period and a display of an automated

*Seated: D. W. Daniel. Standing: J. Waterbury, J. J. Giambrone, H. B. Hulse, and C. A. Kremers*

*At the October meeting of the Western District of the Empire State Section*

low sheet illustrating line flows, equipment motion and equipment arrangement.

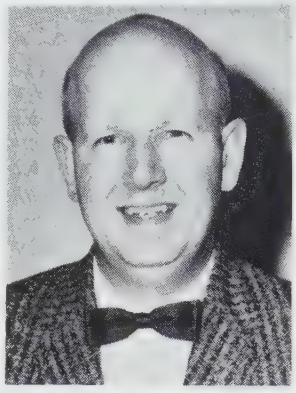
FRANK W. LOREY, *Chairman*

## *Empire State (Western District)*

The first meeting of the Western District for the 1961–62 season was held Oct. 4, 1961. It consisted of a plant tour of the U. S. Gypsum Co. at Oakfield, N. Y. (paper mill and board mill) and a dinner meeting at the Apple Grove Inn, Medina, N. Y. D. W. Daniel, personnel superintendent of U. S. Gypsum Co. spoke on "The Mining of Gypsum and Its 1008 Uses."

Since S. F. M. Maclaren, Penick & Ford, Ltd., has been transferred to their New York Office, R. Zimsmeiter is now Membership Committee chairman and L. B. Timmerman is Membership vice-chairman.

H. B. HULSE, *Chairman*



*H. B. Hulse, chairman, Western District, Empire State Section*

## *Gulf Coast*

### Experiences with Metal Failure Problems in the Pulp and Paper Industry

*W. M. Stephenson, J. R. Nelson, and E. G. Johnson*

**The economic importance of recognizing and solving metal failure problems in the paper industry are considered. Basic types and causes of metal failures are discussed and illustrated. Analytical methods are reviewed and field corrosion measurements briefly considered. Special attention is focussed on the problem of fourdrinier wire life and the three factors which influence it. These were mechanical damage, abrasive wear, and corrosion. None of the three are independent. They must be considered together and solved as one complex problem. Methods available for doing this include personnel education, special attention to mill cleanliness, good operating procedures, and the application of specially designed chemical formulations to reduce wear, maintain drainage, and eliminate corrosion. The potential savings in wire replacement costs, lost production, down time, and labor costs are great enough to justify continued attention.**

IT IS almost impossible to assess accurately the cost of metal failure to American industry. Many plants do not have anyone specifically assigned to the task of monitoring and reducing these problems, and data are sparse. A recent article in *Chemical Processing* magazine (*1*) assigned a figure of 6 billion dollars as the amount lost by all American industry in 1960 due to corrosion alone. No breakdown as to specific industries was given. When metal failures other than corrosion are considered and an attempt is made to include lost production and extra labor costs involved in the downtime to replace or repair metal that has failed, the total dollar loss would be staggering. Fifteen billion dollars might be a conservative estimate.

Pulp and papermaking operations are particularly susceptible to metal failure problems, since their nearly continuous operation makes it difficult to carry out good preventive maintenance programs. Water usage in each mill of 20 million gallons per day or more is common. Therefore, chemical protection of the vast amounts of metal in a papermaking system is hard to achieve. While we cannot assign a dollar value to the metal failure problems in the paper industry, we know the magnitude is great. Each member of this TAPPI group surely is aware of the significance of metal failure in his individual mill.

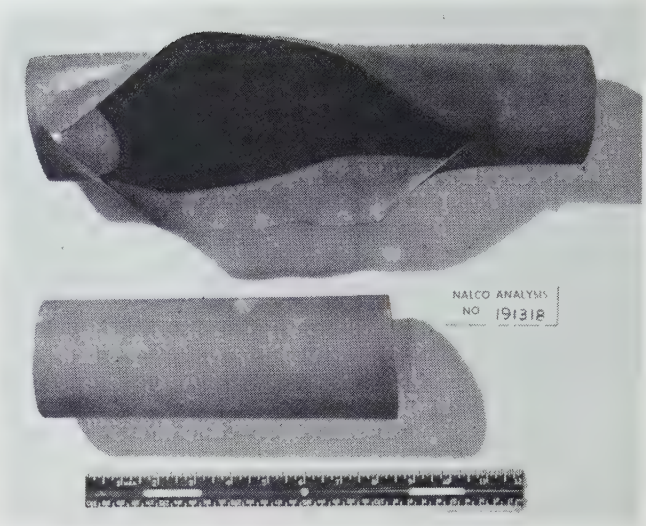



*Fig. 1. Gross photograph of ruptured boiler tube*

Metal failure problems can and do occur throughout the papermaking system—from the woodyard through the power plant and pulp mill to the paper machine and into the converting plants. This discussion will be centered around a very brief description of several general type failures which are common to the pulp and paper industry. It will be focussed primarily on a problem which has commanded a great deal of attention, the failure of fourdrinier wires. Brief descriptions of corrosion measurement techniques, analytical considerations, and metallographic examination procedures will be included.

#### REPRESENTATIVE TYPE METAL FAILURES

Overheating to various degrees is a very common metal failure problem which occurs in power boilers, recovery boilers, and steam generating units. A study of the metal structure at several points in the region of failure will usually yield information about the severity of the overheating. As some metals

*Fig. 2. Martensitic Structure (500×)*

W. M. STEPHENSON, J. R. NELSON, and E. G. JOHNSON, Nalco Chemical Co., Chicago 38, Ill.

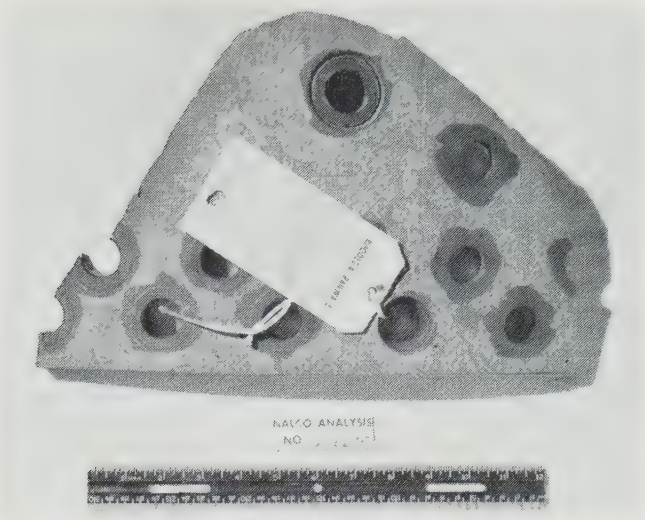


*Fig. 3. Boiler plate—gross photograph*

are heated, structural changes occur which depend on the temperature reached and time exposed. As an example of what can happen, Fig. 1 shows an overheated boiler tube which ruptured violently.

The temperature of this tube had exceeded the upper critical limit before rupture and drastic quenching occurred at the time of rupture through water and vapor issuing from the fracture as shown by the "martensitic" structure in this slide. Figure 2 was prepared from a photomicrograph taken at a magnification of 500×.

Failure of this type in a papermill powerhouse can easily shut down the mill until such time as repairs can be made.

A second type of failure which is often encountered in boilers and can easily occur in kraft digesters is caustic embrittlement. When metal is under stress, contact with a water or liquor containing caustic and small amounts of silica can cause embrittlement if a small leak allows concentration of alkaline solids on stressed surfaces. Figure 3 shows a boiler plate section which appeared sound to the naked eye. It was actually greatly weakened by embrittlement.

Metallographic examination revealed internal damage in the form of cracks in the metal structure. Figure 4 illustrates the type structure encountered in caustic embrittlement cracking. This photomicrograph was taken at an enlargement of 500×.

The use of corrosion-resistant alloys does not always guarantee freedom from metal failure problems. In work done on corrosion-resistant alloys to be used in sea water-cooled heat

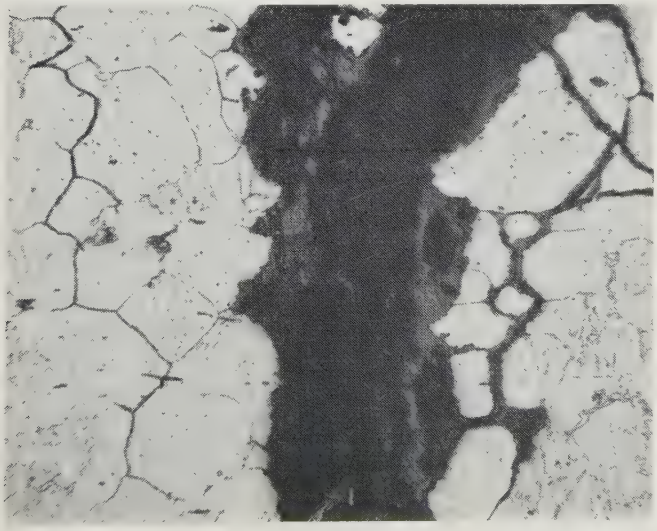


*Fig. 4. Embrittlement cracking (500×)*

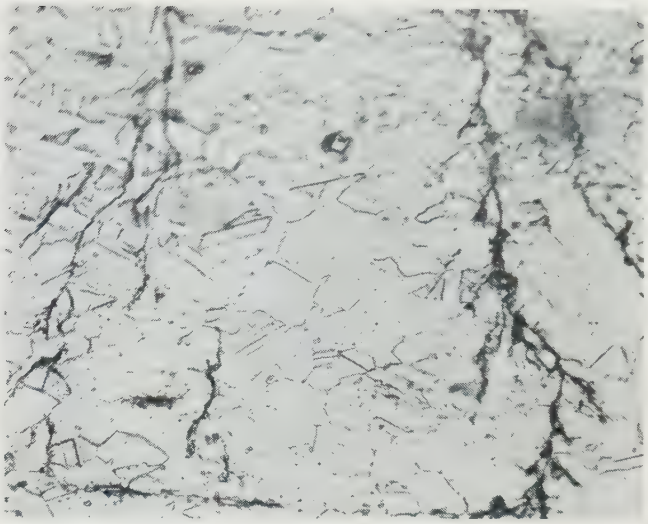


*Fig. 5. Stress corrosion cracking in type 347 stainless steel (500×)*

exchangers in nuclear power plant applications, excessive stress cracking was found in certain alloys. Figure 5 shows stress corrosion cracks originating at the stressed surface of a sample of type 347 stainless steel. The magnification here is 500×.

Erosion of metals can cause severe damage and put equipment out of operation in a short period of time. Figure 6 shows a stainless steel centrifugal pump impeller which has been badly damaged by erosion while pumping a filler clay slurry.

Microbiological attack can occur on nearly any metal surface in a pulp or paper mill where deposits collect or anaerobic conditions exist. Anaerobic bacteria caused the pitting type corrosion attack on the stainless steel coating color tank illustrated in Fig. 7 showing a piece of metal taken from the side wall of the tank. An adequate coating preservation program could have prevented this serious metal loss.

Chemical corrosion can destroy any pipeline or stock line in a mill system if allowed to proceed unchecked. Figure 8 shows a chemical line constructed of ferrous metal which was destroyed by straight chemical corrosion.

These examples constitute only a very brief sampling of the general types of metal damage going on in your mills every day. An awareness of the potential problems is required to be able to effectively combat the problems. Some of the problems can be easily recognized by anyone. More frequently the services of a trained metallurgist are required to ac-

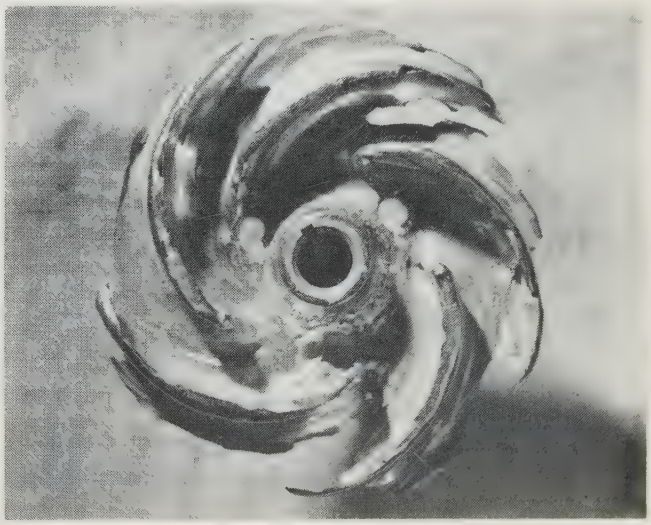


*Fig. 6. Eroded stainless steel pump impeller—gross photograph*

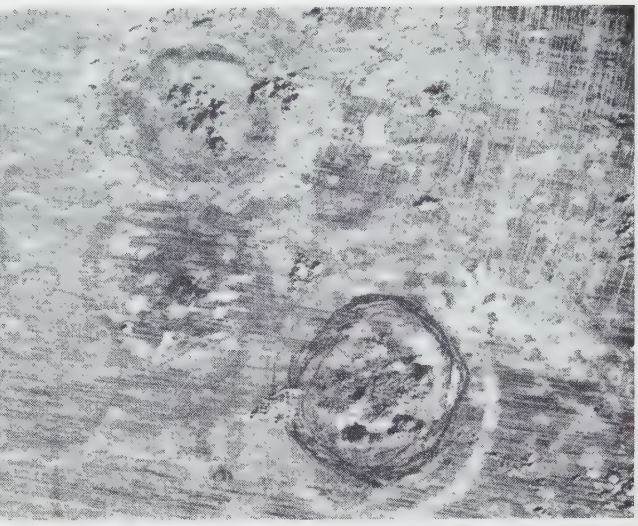


*Fig. 7. Microbiological corrosion on stainless steel coating color tank—gross photograph*

curately diagnose the factors involved and to translate the observations into improvements in operating results.

### METALLOGRAPHIC ANALYSIS—A KEY TOOL IN EXAMINATIONS OF METAL FAILURES

All of the pictures shown, as well as those to be shown later, were prepared in our metallographic and photographic laboratories. In over 35 years of dealing with metal problems and the protection of metals, Nalco has gained a large amount of experience in examining failed metal, ascertaining the causes for the failures, and recommending solutions to the problems. The metallographic and associated analyses are key tools in doing this. A brief discussion of the procedures and techniques employed would be helpful at this point. Any combination of, or all of, the following steps may be included as necessary.

1. The first step in a metallographic examination is photographing the sample from various angles to record the exact appearance of the failure in the sample received. Then, trained metallurgists examine the sample to determine what steps should follow.

2. The sample is next examined under low and high magnification with a binocular microscope to get an accurate three dimensional view of the characteristics of the failure. If necessary, the sample can be sent to an independent laboratory equipped for Magnaflux and Magnaglo examination to accurately locate surface and internal imperfections or failures.

3. Then, several small samples will be cut, molded in plastic, ground, finished, and etched in order to examine and photograph the metal structure under high magnification through a special metallographic microscope. For extremely high magnifications and study of very minute characteristics of the metal, the electron microscope may be used.

4. Specimens of the normal metal may be subjected to dynamic testing in specially designed corrosion, abrasion, and embrittlement testing units to duplicate the problem and confirm analysis results.

5. If the sample is corroded, scaled, or contains a deposit of any kind, this will be removed and analyzed in the analytical laboratories using chemical, photometric, infrared, chromatographic, and x-ray techniques.

6. Samples of the process water, solution, suspension, slurry, or dispersion which contacted the metal during its service will be analyzed completely by standard chemical or instrumental analytical methods. When all of the data are collected and properly interpreted in light of operational information, a thorough understanding of the causes of the problem will be apparent. From an understanding of the problem, recommendations for solving it can be intelligently made.

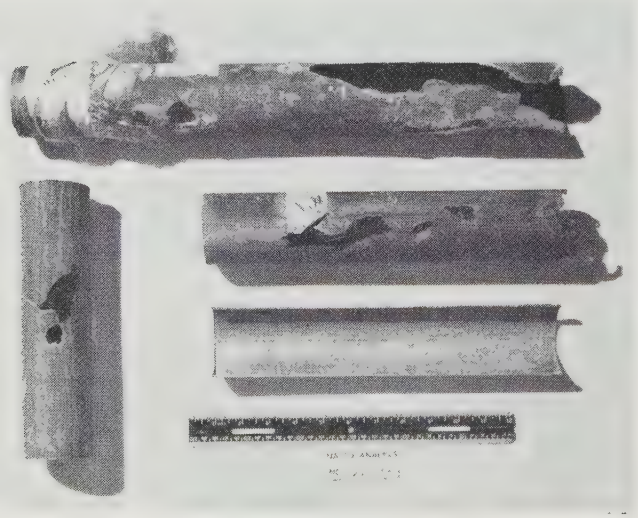


*Fig. 8. Chemical corrosion of chemical line—gross photograph*

### CORROSION MEASUREMENTS IN THE FIELD

The nature of the pulping and papermaking operations makes corrosion of equipment one of the major metal failure problems in your industry. Many authors have defined the various types of corrosion and the factors which influence corrosion rates. For purposes of this discussion, it suffices to say that corrosion is accelerated by low pH, high temperature, high total acidity, high dissolved solids contents in process waters and white waters, corrosive dissolved solids and gases, vigorous aeration, and the repeated abrading off of protective films or corrosion products. Microbiological activity can also cause corrosion. All of these conditions for promoting corrosion exist in a paper mill and are estimated to cause losses of approximately 30 to 90 million dollars annually. Metallographic analytical techniques are extremely valuable in understanding and solving corrosion problems, but field methods to measure corrosion and monitor process changes and their effect of corrosion in operating equipment are frequently necessary. Available field methods were discussed by Nelson in an article appearing in the July 24, 1961, issue of *Paper Trade Journal* (*2*). Basically, these methods include inspection of the equipment during periods when it is not in operation, review of maintenance cost records, chemical analyses of process water, installation and use of corrosion coupons throughout the system, and direct measurement with electrical resistance probes. Equipment inspection is perhaps the best way of ascertaining the damage being done to the metal in question. However, it is frequently impossible to adequately inspect an operating unit, and damage may be hidden in inaccessible areas. Months or years may pass before damage is detected in this way and then it is too late to prevent it. Corrosion coupons provide a more rapid means of measurement, normally between 15 and 60 days. Coupon data are useful for comparing process changes, but are limited to "average" values and will not reflect rapid or frequent operational changes. Within the last ten years, electrical resistance probes have come into use to provide a rapid measurement of corrosion rates. Several probes can be installed throughout stock and white water systems and all measurements then made with one instrument without interrupting production. The order of magnitude of corrosion rates can be measured in a few days, or hours in some cases. The influences of changes in furnish, water, pulp washing, and stock preparation can be detected and measured as they occur. Immediate steps can then be taken to properly protect equipment before irreparable damage is done.

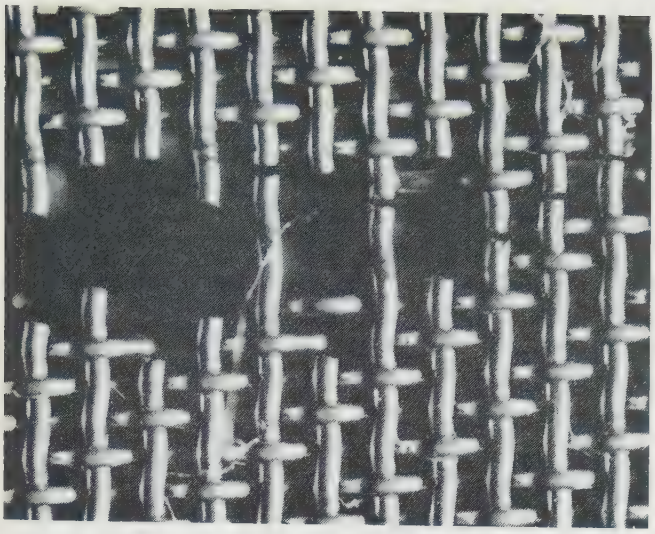


*Fig. 9. Tear and cracked shute wires (12×)*

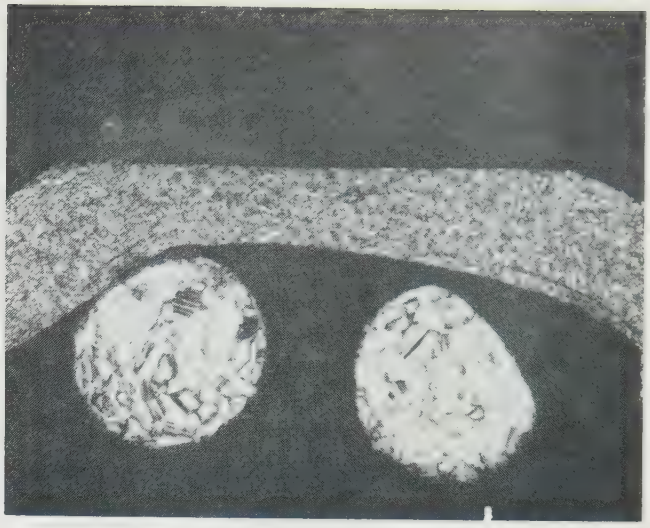


*Fig. 11. Worn warp wire (250×)*

#### METALLOGRAPHIC EXAMINATIONS AND FOURDRINIER WIRES

With many years of experience in metal failure problems, we have also investigated what is probably one of the major metal failure problems in the paper industry today—the problem of fourdrinier wire life. The economic situation which has existed over the last few years, a more acute awareness of operating costs, austerity programs, and the trend to bigger and faster paper machines have focussed attention on the large savings which can be realized by achieving the maximum life out of each fourdrinier wire installed. Samples we have received for metallographic analysis consistently show good quality in fourdrinier wires. In those examined, no evidences of faulty material or defective manufacture have been detected. This means that when a wire gives less than satisfactory life, the situation can usually be corrected within the paper mill.

#### ECONOMICS OF WIRE LIFE IMPROVEMENT

The Lindsay Wire Weaving Co. of Cleveland, Ohio has quoted wire costs as high as $2.80 per ton in some high speed newsprint mills and lists as an average for all newsprint mills a figure somewhere above $1.30 per ton (*3*). Data collected during the last six months on wire costs in the southern kraft industry indicate that $0.65 to $1.25 per ton are fairly typical values for mills in this area. An average annual increase of 50% in the life of fourdrinier wire will reduce costs by one-third, while doubling the wire life will cut the costs in half. This kind of cost savings is easily within the realm of possibility for those mills or machines where the average wire life is 15 days or less. In addition to the direct savings in wire replacement costs, the reduction in downtime for wire changes and the reduction of labor costs for the extra manpower involved in wire changes can make even relatively short wire life extensions quite attractive to the papermaker.

#### FACTORS IN REDUCED WIRE LIFE

Most fourdrinier wires are removed from service long before they have completed a normal tour of duty. One of the major causes for early removal is mechanical damage suffered during installation or operation on the machine.

1. Ridges, wrinkles, or creases can result from rough or careless handling during transit, storage, or installation. Dirt, grit, or stock on machine surfaces or stringing poles will cause dents, jams, or pimples in the wire during installation. Scrupulous cleanliness and thorough education of mill personnel in wire handling can eliminate this cause of damage.
2. Damage during operation frequently occurs as stock or other foreign material is forced between the wire and one of the outside rolls. This causes a dent or downward jam which will quickly wear into a hole. Plugged or filled wires can easily suffer this type damage due to inefficient stock removal. Material on overhead crane rails, the headbox, catwalks, or stairways near the wet-end can be blown or washed onto the wire causing dents or holes.
3. Upward dents or pimples result when hard stock or for-

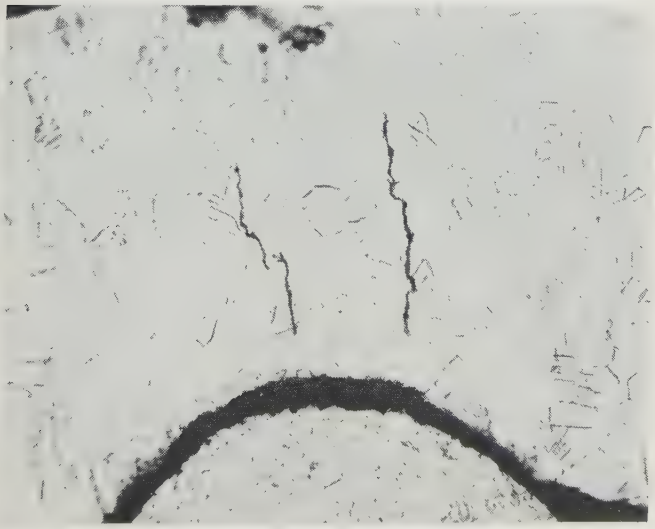


*Fig. 10. Intergranular cracking in brass shute wire (500×)*

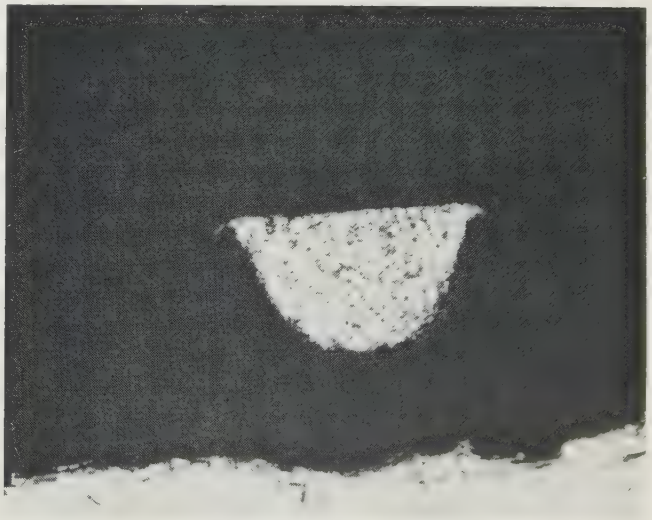


*Fig. 12. Cross section worn warp wire (250×)*

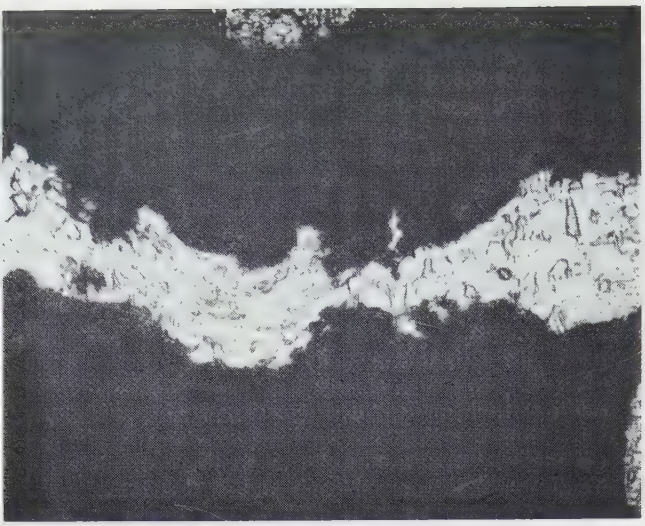


*Fig. 13. Longitudinal section of badly corroded shute wire (250×)*

eign material is forced between the wire and an inside roll. Such material can drop off the suction boxes, trays, shower pipes, or machine frame. These upward pimples cause light spots in the sheet and may in extreme cases hang on the slice and cause a tear.

4. Ridges, wrinkles, or creases which are responsible for many short wires can also result from misalignment of a roll which results in whipping or vibration, unbalance in a roll, improper tension in the wire, a vibrating deflector or a build up of pitch, slime, stock or foreign material on the flat boxes or deflectors. Some wires are wrinkled when cold fresh water replaces hot white water in the showers during a shutdown.

5. Sudden changes in flat box or couch vacuums, variations in the motor load on drive rolls, or previously undetected splits and holes can cause wires to tear off the machines. Flat box oscillators stopped or running in unison can cause the wire to hang on the boxes and tear. Failure of the automatic guide can allow the wire to run off. People or objects can fall or run into wires.

Figure 9 illustrates one type of damage just discussed. The tear in this wire occurred when a misalignment of some part of the machine caused the wire to whip. Intergranular cracking of the brass shute wires resulted.

Figure 10 illustrates the damage to the metal structure of the cracked shute wires just shown. Here, intergranular cracking which weakened the brass metal structure is evident. This photomicrograph was taken at 500×.

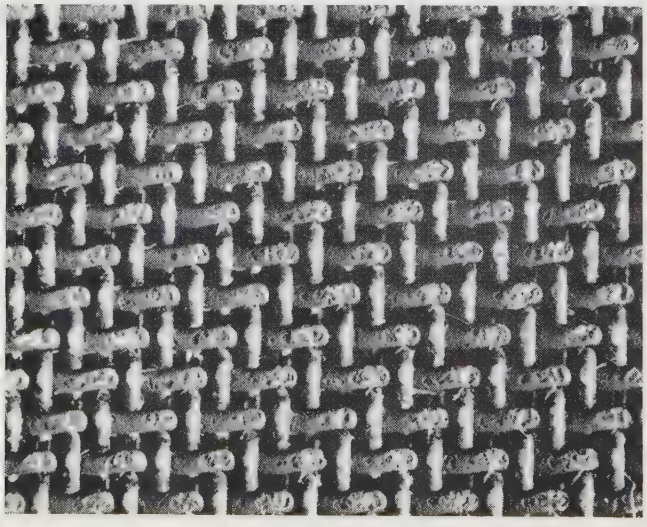


*Fig. 14. Pitted wire—sulfide attack (12×)*

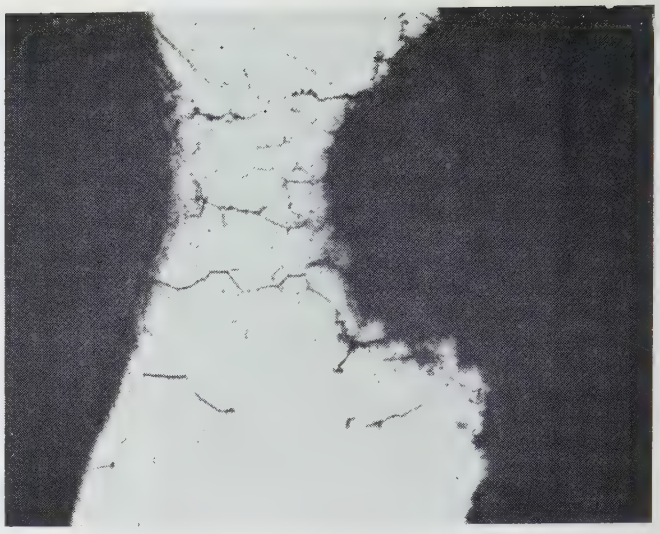


*Fig. 15. Transgranular cracking as a result of corrosion fatigue—shute wire (400×)*

The primary reason for wire replacement is wear. The type of alloys used in the wire, the mesh, temperature, pH, chemical additives, fillers, grade of paper produced, and general machine condition all influence wear on the wire.

1. Abrasive wear occurs on the suction boxes, deflectors, forming board, deckles, and on the couch roll.

2. Sand or grit in the white water, the use of abrasive fillers, and high temperatures which lower the viscosity of the water will accentuate the wear rate.

3. Filling of the wire by stock or furnish chemical deposits, pitch or corrosion products, and the reduction in open area caused by wear necessitate higher flat box vacuums to remove water from the sheet. This increases the wear at the boxes.

4. Poor machine design or poor maintenance often contribute to wear. Running the wire around sharp radii can cause wear hardening and fatigue failure.

5. Allowing the wire to run over dirty or grooved suction box covers or faulty setting of the slice and deckles will greatly increase wear.

Figure 11 shows a longitudinal view of a worn warp wire at an enlargement of 250×. Note the semitwill weave of this particular wire.

Figure 12 illustrates the wear on a warp wire in a transverse view at an enlargement of 250×. Note the burring which reduces the open area of the wire and slows its effective drainage.

The third classification of factors which influence wire life is corrosion. Authorities differ considerably in their opinions

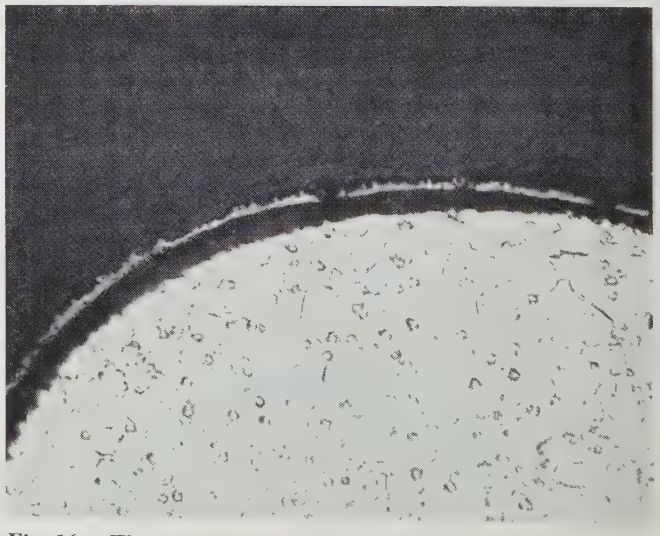


*Fig. 16. Warp wire cross section showing layer of corrosion products (500×)*

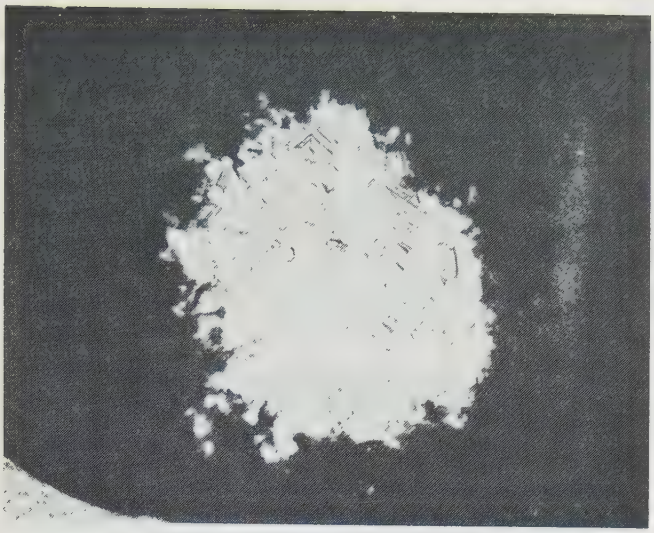


*Fig. 17. Corroded shute wire—cross section (250×)*

concerning the severity of corrosion problems in reducing wire life. Some mills are constantly troubled with corrosion, others have unrecognized problems, and still others may operate for long periods of time without encountering it. When it does occur, it can drastically shorten wire life. In this discussion, consider that wire life is influenced by a combination of damage, wear, and corrosion factors and where corrosive conditions do exist that they contribute considerably to increased wear rates. Most white waters are acid, contain high concentrations of dissolved solids, and are vigorously aerated in the papermaking process. Materials used in the furnish may increase corrosiveness. They may enter the system from the pulp mill or may be present in the process or raw water. High temperatures are frequently added to the above conditions to produce white water with an aggressive corrosion potential. The types of corrosion encountered in fourdrinier wires is similar in nature to that found in tanks, pipelines, white water lines, stock lines, boilers, cooling towers, etc. Metallographic analysis will show the exact nature of the corrosion and its relationship to the wear and damage factors involved. Even though the brass and bronze alloys normally used for the shute and warp wires respectively are quite similar, their resistance to corrosive attack is quite different. The brass shute is generally more susceptible than the bronze warp and provides some anodic protection to the warp.

Figure 13 shows a very badly corroded shute wire at a magnification of 250×. The warp wires from this particular sample were almost free from attack even though the machine operated at a high temperature and at very low pH's.

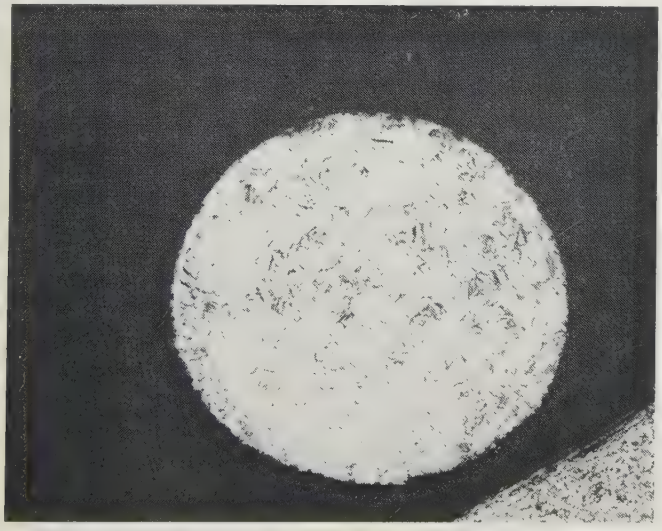


*Fig. 18. Protected shute wire—cross section (250×)*

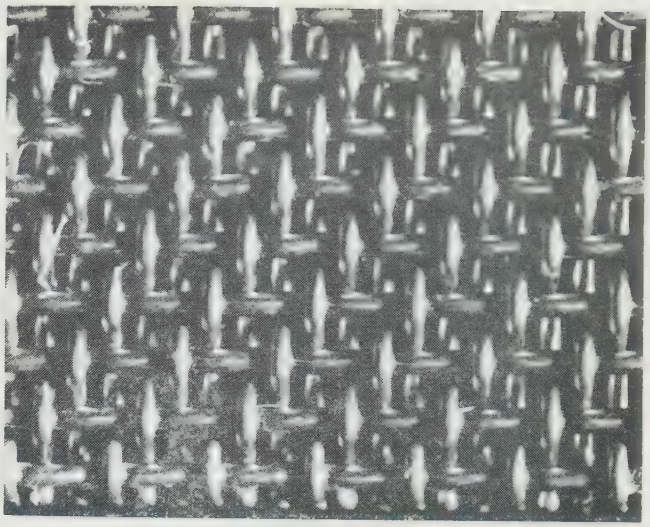


*Fig. 19. Protected wire (12×)*

A pitting-type attack is frequently noted under sulfide deposits. This type attack is especially prominent in areas where the hydrogen sulfide content of the fresh water is high. Even when the sulfides in fresh water are oxidized with chlorine, typical sulfide pitting attack is frequently observed.

Figure 14 shows a section of wire at a magnification of 12× in which the brass shute knuckles are severely pitted. X-ray analysis of the deposit covering the wire showed it to be primarily copper sulfide. The fresh water supply at this particular mill is high in hydrogen sulfide content.

Transgranular cracking is often associated with corrosion and fatigue. As flexing and fatigue break down surface films of corrosion products, deep penetration, and concentrated local attack is likely. Corrosion and fatigue also play important parts in increasing the wear rate on the warp wires. When the relatively soft corrosion products are flexed or abraded off, a clean fresh surface is exposed which can corrode rapidly. Alternate corrosion and abrasion in this manner can accelerate wear rates and shorten wire life.

Figure 15 demonstrates transgranular cracking of a shute wire which resulted from alternate corrosion and fatigue. This photomicrograph was taken at a magnification of 400×.

Figure 16 illustrates at a magnification of 500× one of the few samples in which warp corrosion has been definitely identified. Please note the adherent layer of corrosion products on the surface of the warp wire. It is this layer which easily abrades off and increases the rapidity of warp wear.

### INCREASING FOURDRINIER WIRE LIFE THROUGH PROTECTION

To repeat, wire life is influenced by *damage, wear,* and *corrosion factors.* The first step in increasing wire life is reducing damage. Careful attention to handling and operating procedures and scrupulous cleanliness on and around the machine will minimize damage to the wire. Good slime control and pitch control programs will reduce build-ups of foreign material on the machine and further reduce damage factors. Wear and damage can both be reduced by a thorough program of preventive maintenance. Careful screening of stock and the use of less abrasive fillers will reduce abrasive wear on the suction boxes. Prevention of wire plugging or filling will maintain effective drainage on the wire and eliminate the need to increase vacuums on the flat boxes. Protection against corrosion can be obtained by increasing operating pH's and reducing dissolved solids in the white water to levels which will reduce the corrosiveness of the white water and still be consistent with good papermaking practices. Improved pulp washing, efficient water treatment, and careful selection of furnish chemicals contribute to a reduction in corrosion. The high temperatures and vigorous aeration encountered in most machine systems can only rarely be avoided.

## CHEMICAL FORMULATIONS TO INCREASE FOURDRINIER WIRE LIFE

In the last few months, several chemical formulations to improve wire life have been offered to the paper industry. Dramatic wire life extensions of 50 to 100% have been demonstrated. These chemicals can be used most efficiently to supplement good operating practices as previously discussed. To do a really effective job, a wire life extender should be formulated of several compatible ingredients to perform at least three functions:

1. The chemical should film the wire to prevent pitch, fines, chemicals, and other deposits from adhering tightly. The wire showers can then clean the wire more efficiently and effective drainage can be maintained. A slight amount of dispersancy aids in this function.
2. The extender should also lightly lubricate the wire to reduce abrasive wear, but not produce slip on the couch and drive rolls.
3. The third important function of a wire life extender is to eliminate corrosion on the wire.

In order to perform these important jobs, a formulation containing several ingredients is necessary. However, the formulation should be considered and applied as a *wire life extending chemical* rather than as an ingredient in another operational chemical to be applied in some other part of the system. This is because the problem is centered at the machine where direct attention is necessary.

The next series of figures illustrate what has been observed in metallographic examinations of failed wires, without and then with wire life extending formulations. Figure 17 shows a badly corroded shute wire cross section at a magnification of 250×.

Figure 18 shows a shute wire from the same paper machine when a wire life extender was being used. This wire ran under the same operating conditions as the wire just shown, but was protected with a wire life extending chemical. This second wire ran approximately 75% longer than the first one.

Figure 19 shows a view at 12× of the wire from which the protected shute was taken. Even though the mill involved had a history of short wire life due to wear and corrosion, this wire which was protected with a wire life extender shows absolutely no corrosion and had less wear than normal wires even after running 75% longer. The wire was ultimately removed because of mechanical damage, a hole caused by a pimple in the wire, and not because it was worn out.

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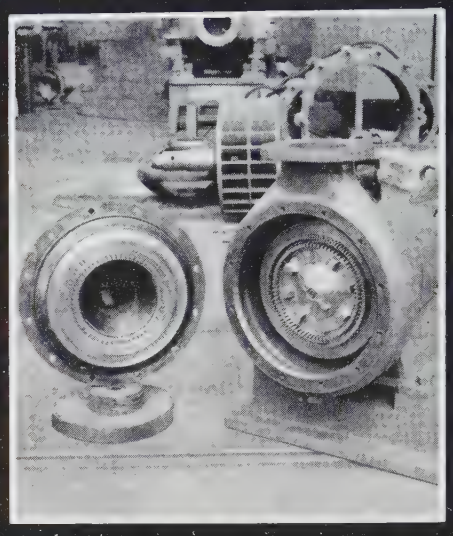
Presented at a meeting of the Gulf Coast Section of TAPPI.

The magnifications specified on the photographic enlargements are those of the original 9 by 11 cm. photomicrographs. The actual magnification in these photographs is less than indicated.


ultra sonics in the paper industry



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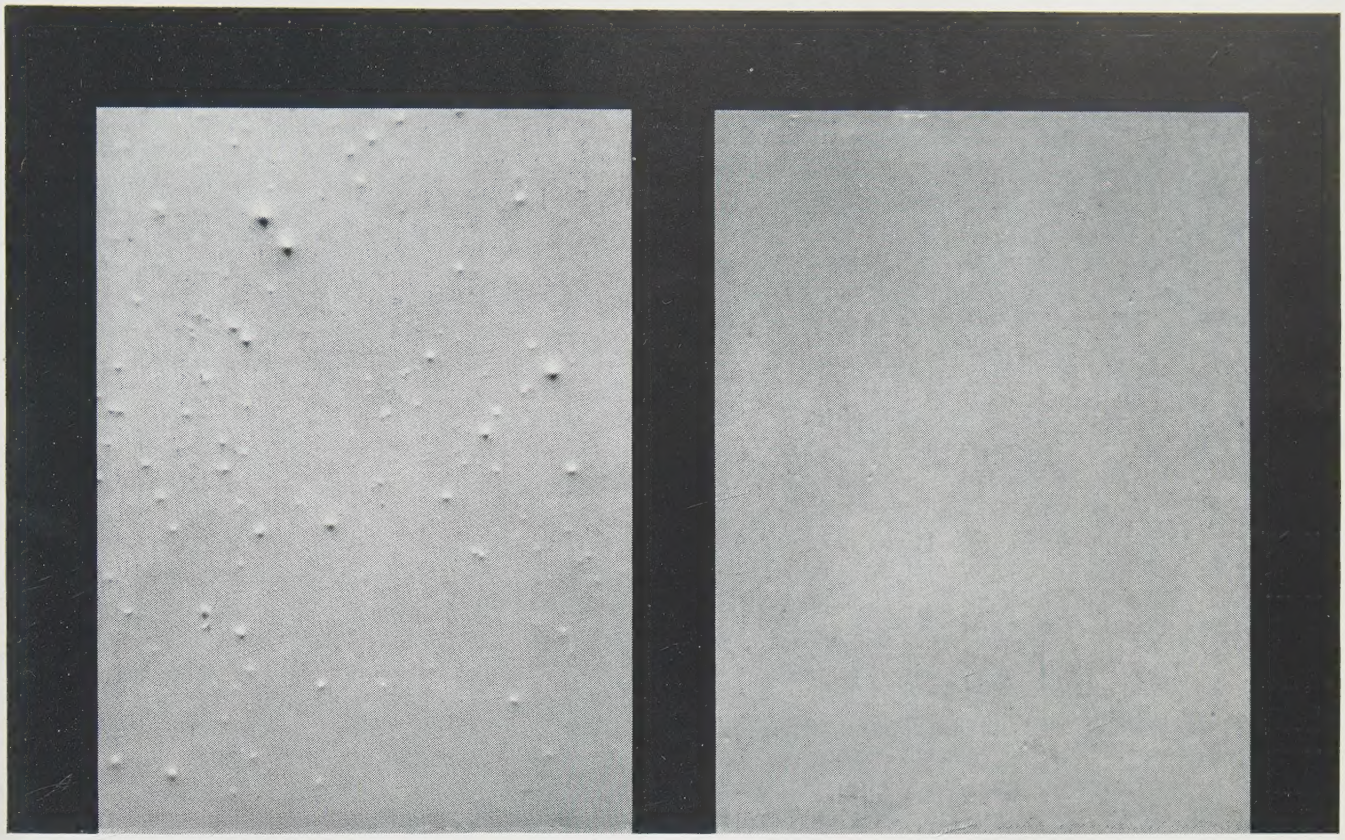

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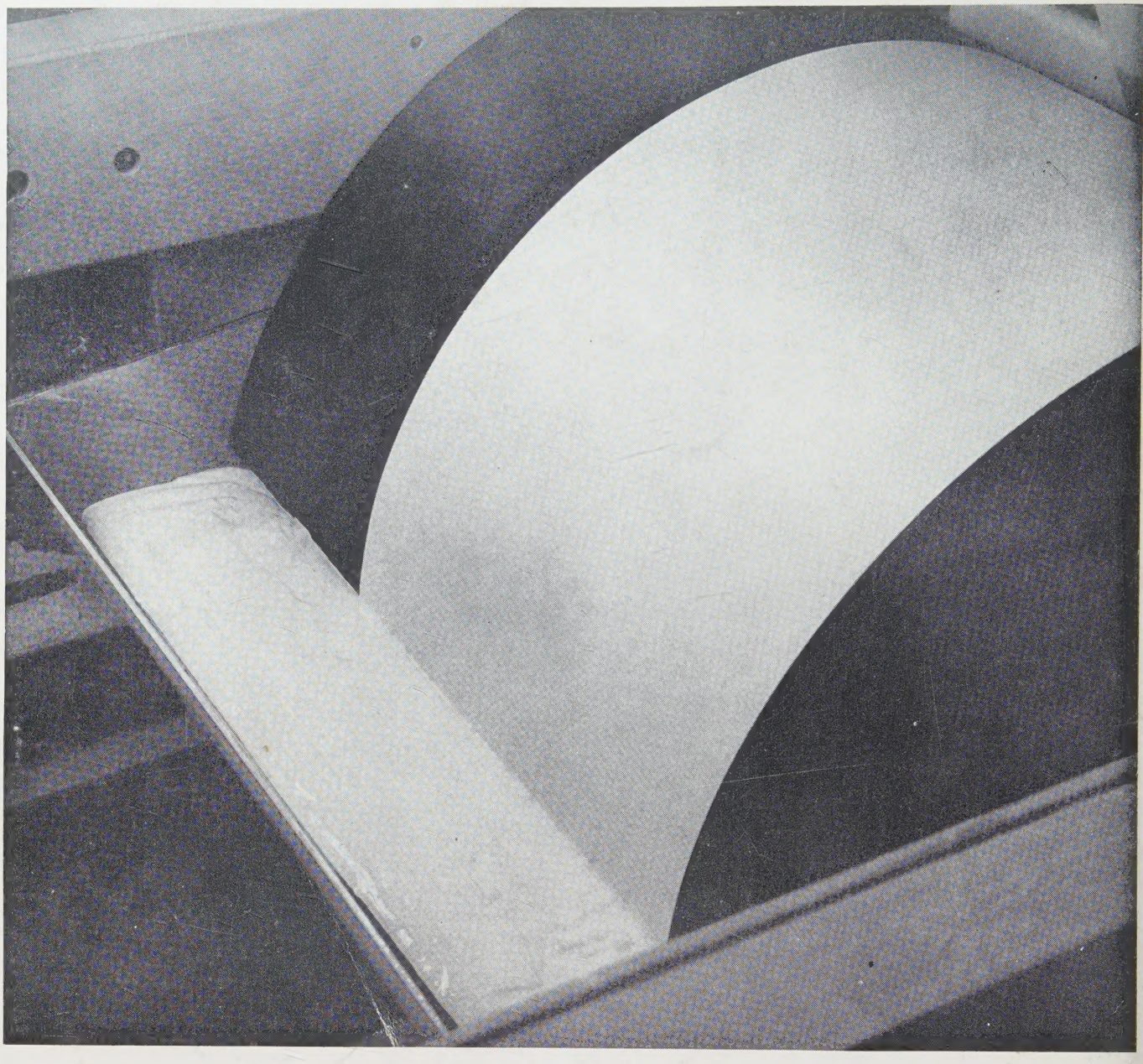

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